Sampling and Analysis Plan/Quality Assurance Project Plan
2014 Sampling Events

Upper Animas Mining District
Gladstone, San Juan County, Colorado

Prepared For:
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Ecosystem Protection and Remediation – Program Support
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List of Abbreviations and Acronyms

ARSG	Animas River Stakeholder Group
BLM	Bureau of Land Management
CA	Corrective Action
CDPHE	Colorado Department of Public Health and Environment
CFR	Code of Federal Register
CLP	Contract Laboratory Program
COPEC	Contaminant of Potential Ecological Concern
DO	Dissolved Oxygen
DOC	Dissolved Organic Carbon
DQA	Data Quality Assessment
DQO	Data Quality Objectives
DRMS	Division of Reclamation, Mining and Safety
DS	Decision Statement
DSR	Data Summary Report
EDI	Equal Discharge Increment
ESAT	Environmental Services Assistance Team
EPA	Environmental Protection Agency
gpm	Gallon per minute
GPS	Global Positioning System
HDPE	High-Density Polyethylene
LCS	Laboratory Control Spikes
LIMS	Laboratory Information Management System
MDL	Method Detection Limit
MMI	Multi-Metric Index
MS/MSD	Matrix Spike/Matrix Spike Duplicate
OSHA	Occupational Safety and Health Administration
PE	Performance Evaluation
PSQ	Principal Study Question
QA/QC	Quality Assurance/Quality Control
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QMP	Quality Management Plan
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SAP	Sampling Analysis Plan
SGC	Sunnyside Gold Corporation
SOP	Standard Operating Procedures

STIL	Stream Temperature Intermittency Loggers
TAL	Target Analyte List
TMDL	Total Maximum Daily Loads
USFS	United States Forest Service
USFWS	United States Fish and Wildlife Service
USGS	United States Geological Survey

A.3 Distribution List

The following is a distribution list of personnel that will receive a copy of the Sampling and Analysis Plan (SAP)/Quality Assurance Project Plan (QAPP) for the sampling events scheduled in 2014 at the Upper Animas Mining District. Agency and/or contractor affiliations are also listed for each individual.

Paula Schmittdiel	U.S. EPA
Steve Way	U.S. EPA
Dan Wall	U.S. EPA
Nicole Plescia	U.S. EPA
Peter Butler/William Simon	ARSG
Kay Zillich	BLM/USFS Project Manager
Craig Gander	CDPHE Project Manager
Steven Auer	ESAT Field Task Lead
Mark McDaniel	ESAT Manager

A.4 Project/Task Organization

The following is a list of involved personnel, respective agency/contract affiliation, and general responsibilities.

Managers:

Paula Schmittdiel	USEPA	Remedial Project Manager
Steven Way	USEPA	On-Scene Coordinator
Dan Wall	USEPA	Task Order Project Officer/ QA Approving Official
Mark McDaniel	ESAT	ESAT Team Manager

Field Team:

Steve Auer	ESAT	Field Task Lead
Ryan Bahnfleth	ESAT	Field Support
Leslie Christner	ESAT	Field Support
Sherry Skipper	USFWS	Field Support
Robyn Blackburn	USFWS	Field Support
Lisa Richardson	BLM	Field Support

Laboratory Group:

Scott VanOvermeiren	ESAT	Inorganic Task Lead
Scott Walker	ESAT	Analytical Support

Organizational Chart

SAP/QAPP Development
Steve Auer (ESAT)

Project Managers
Paula Schmittdiel (USEPA Remedial Project Manager)
Steven Way (USEPA) On-Scene Coordinator

SAP/QAPP Implementation
Field Team Leads
Steven Auer (ESAT)
Dan Wall (USEPA)

Field Team

Steven Auer (ESAT)
Leslie Christner (ESAT)
Ryan Bahnfleth (ESAT)
Kelsey Bartling (ESAT)

EPA Approving Official

Dan Wall (USEPA)

Task Order Project Officers

Dan Wall (USEPA)
Nicole Plescia (USEPA)
Don Goodrich (USEPA)

ESAT QA Manager

Nikki MacDonald

ESAT

Mark McDaniel (ESAT Team Manager)
Steven Auer (Senior Field Task Lead and SAP/QAPP Development)

Stakeholders

United States Environmental Protection Agency
United States Geological Survey
United States Forest Service
United States Fish and Wildlife Service
Bureau Land Management
Colorado Department of Public Health and Environment
Colorado Division of Mining and Safety
Animas River Stakeholder Group
San Juan County

Region 8 Laboratory Group
Scott VanOvermeiren (ESAT Analytical Lead)
Scott Walker (ESAT Chemist, Laboratory Quality Assurance)

A.5 Problem Definition

Cement Creek, the receiving stream for the discharge of the metals-contaminated water and sediments from mines in the Cement Creek watershed, including the American Tunnel, Red & Bonita, Mogul, and Gold King Level 7 is unable to support aquatic life. Cement Creek is a major contributor of metals and acidity to the Animas River. The river has goal-based cold water aquatic life standards established by the State of Colorado Water Quality Control Division (CDPHE, 2013).

The U.S. Environmental Protection Agency identified a number of data gaps, including lack of sufficient data on the current state of contamination in biotic and abiotic media; effects on aquatic organisms and exposure to wildlife receptors through the food chains; and identification of a possible source of metal contamination upstream of sample locations A56. In addition, possible sources of contamination may exist further downstream of A72 in or around the proximity of the Chicago Basin area. In order to address these data gaps, this SAP will present a plan to collect sediment, surface water, macroinvertebrate and fish tissue samples for laboratory analysis. This data collection effort will assist in further understanding of the current state of contamination in abiotic and biotic media and impacts on the aquatic ecosystem and potential risks to wildlife receptors through the food chain and will accomplish the following:

- Determine the seasonal and temporal metals loads in the Cement Creek watershed and Animas River

- Determine the concentrations of metals in the Animas River water immediately before, during, and after high-flow conditions

- Determine the nature and extent of sediment metal contamination in the Animas River between Howardsville and Bakers Bridge

- Determine the level of contaminants of potential ecological concern (COPECs) in fish and invertebrate tissues to refine risk estimates of food chain exposures of wildlife receptors.

A.5.1 Introduction

The EPA, in cooperation with participating stakeholders will be sampling Cement Creek and the Animas River as part of a Remedial Investigation (RI). Mobilization and watershed sampling activities are scheduled to be performed from April 2014 to October 2014.

This SAP has been prepared in accordance with the EPA "*Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA QA/G-4)*, *Requirements for Quality Assurance Project Plans (QA/R-5)* and the "*Guidance for Quality Assurance Project Plans (EPA QA/G-5)*, (EPA 2006; EPA 2001; EPA 2002). This SAP is designed to guide field work that will include the collection of surface water, sediment, and biota samples as well as flow measurements, including field Quality Assurance/Quality Control (QA/QC) samples.

Laboratory analyses will include total and dissolved Target Analyte List (TAL) metals, hardness (calculated from calcium and magnesium concentrations in the dissolved ratio) anions, alkalinity, and dissolved organic carbon (DOC). All samples will be analyzed either directly or indirectly (by means of subcontractors) through the ESAT Region 8 Laboratory. Field-collected measurements of aqueous samples will include temperature, pH, specific conductivity, dissolved oxygen (DO), and flow measurements.

Sampling procedures will adhere strictly to those outlined in the attached Environmental Services Assistance Team (ESAT) Region 8 Standard Operating Procedures (SOPs). Deviations from the SAP will be documented in the site dedicated field notebook and reported in the Sampling Activities Report. Deviations that result in major modifications to the SAP will be noted and incorporated into all addenda to the SAP, which will be followed as applicable for subsequent sampling events. A brief addendum to the SAP will be created for each subsequent sampling event by updating relevant figures, tables, and attachments. Data obtained from these investigations will be used in accordance with the provisions outlined in the Data Quality Objectives (DQOs).

A.5.2 Background

The discovery of gold and silver brought miners to the Silverton area and Animas Mining District in the early 1870's. The discovery of silver in the base-metal ores was the major factor in establishing Silverton as a permanent settlement. Between 1870 and 1890, the richer ore deposits were discovered and mined to the extent possible. Not until 1890 was any serious attempt made to mine and concentrate the larger low-grade ore bodies in the area. By 1900, there were twelve concentration mills in the valley sending products to the Kendrick and Gelder Smelter near the mouth of Cement Creek. Mining and milling operations slowed down circa 1905, and mines were consolidated into fewer and larger operations with facilities for milling large volumes of ore. After 1907, mining and milling continued throughout the basin whenever prices were relatively favorable.

Gladstone, located about eight miles upstream of Silverton on Cement Creek, is the site of an historic mining town developed in the 1880s commensurate with the onset of mining in the surrounding area. The town was the central location and railroad terminus for the milling and shipping of mine ores from the surrounding three-square-mile valley.

The town declined in the 1920s and no remnants of the town remain. By the 1970's only one year-round productive mine (Sunnyside Mine) remained in the county. This mine ceased production in 1991, and has since undergone extensive reclamation efforts. The Gold King Mine's permit with the Division of Reclamation, Mining and Safety (DRMS) is currently in inactive status; however, landowners hope to rehabilitate the mine. Both the Sunnyside and Gold King properties were partially accessed through the American Tunnel that has its portal in Gladstone.

Previously the American Tunnel drained as much as 1,600 gallons per minute (gpm) of water from the mines. A lime feed and settling pond type treatment facility was constructed in Gladstone in 1979 by Standard Metals Corporation. Water discharging from the American Tunnel was treated as required by the water discharge permit. The facility operations and mine ownership was later transferred to the Sunnyside Gold Corporation (SGC). Under jurisdiction of a court consent decree to terminate their discharge permit, SGC installed several bulkheads within the Sunnyside Mine that greatly reduced the amount of discharge from the American Tunnel. Seventy to one hundred gpm continue to discharge presumably from near surface groundwater. All terms of the consent decree were met by SGC in 2002.

In January 2003 the treatment facility, operations, and permit were transferred to the Gold King Mines Corporation. The settling ponds were deeded to the San Juan Corporation by SGC prior to the lease between the Gold King Mines and San Juan Corporations. The treatment facility continued to treat the remaining American Tunnel discharge and the Gold King Mines discharge until September 2004. The San Juan Corporation required SGC to reclaim the four settling ponds (completed in 2005) following termination of the San Juan Corporation and SGC lease. The Gold King Mines Corporation was subsequently evicted and the balance of the Gold King Mines Corporation land was acquired by the San Juan Corporation as the lien holder. The American Tunnel portal reclamation and removal of some out buildings were completed in 2006. The Bureau of Land Management (BLM) manages land associated with the American Tunnel portal and vicinity; however, the San Juan Corporation owns the majority of the land surrounding the portal.

Numerous historic and now abandoned mines exist within a two-mile radius of Gladstone. They include: the Upper Gold King 7 Level, American Tunnel, Grand Mogul, Mogul, and Red and Bonita, Evelyne, Henrietta, Joe and John, and Lark mines. Some of these mines have acid mine drainage that flows between 30 and 300 gpm directly or indirectly into Cement Creek and eventually into the Animas River, the confluence located about eight miles downstream of Gladstone. The Animas River Stakeholder Group (ARSG), BLM, DRMS and private stakeholders have completed remediation projects at the Evelyne, Henrietta, Joe and John, and Lark mines. The remaining sites located in the Cement Creek drainage that will be the focus of these sampling efforts include the American Tunnel, Grand Mogul, Mogul, Red and Bonita, and the Upper Gold King 7 Level.

The EPA along with the ESAT, United States Fish and Wildlife Service (USFWS), and United States Geological Service (USGS) performed three sampling events in 2012 to 2013 to add to previously collected data. Over the three events, surface water, sediment, macroinvertebrates, stream flows and real-time water quality parameters were collected.

A.6 Project/Task Description

Sampling events will be conducted in 2014 to evaluate the extent of metals contamination in surface water and sediment in the Cement Creek drainage and the Animas River between Howardsville and Bakers Bridge. Macroinvertebrate and fish populations will be surveyed and tissue will be collected during both surveys for metal analyses. Data generated from the sampling events will be used in accordance with the established DQOs (Section A.7.2) and will provide data to support a Baseline Ecological Risk Assessment. The following data will be collected during the events:

- Real-time field water quality parameters – pH, conductivity, dissolved oxygen, temperature, and Global Positioning System (GPS) locations (if needed)
- Stream flows – using SonTek™ flow meters, flumes (where necessary), and USGS gauging stations
- Surface water samples will be collected at predetermined sampling locations in streams and adit discharges for measurement of dissolved and total recoverable metals, alkalinity, anions, and DOC.
- MiniSippers will be deployed at seven locations for the collection of a daily integrated water sample for the period of April through July 2014. A collocated grab water sample will be collected at stations A72 and A68 approximately once a week while MiniSippers are deployed (See Section B.2.2)
- Stream Temperature Intermittency Loggers (STILs) will be deployed at ten locations in the Animas River for logging of water conductivity. STILs will be placed to bracket the confluence of Ten Mile Creek, No Name Creek, Ruby Creek, Pidgeon Creek, and Needle Creek and the Animal River. Exact locations for placement of the STILs will be determined in the field based on terrain and accessibility (See Section B.2.2)
- Sediment samples – total recoverable metals and mercury
- Pore water samples – dissolved metals
- Macroinvertebrate – total recoverable metals, mercury, and demographic analysis
- Fish population and total recoverable metals and mercury analysis in tissue

- Photolog – site photographs will be collected for all sampling locations

A.7 Quality Objectives and Criteria

This section discusses the DQO process and how it was applied to this study. Specific areas addressed include: the planning team and stakeholders; DQOs; and parameter metrics such as precision, accuracy, representativeness, completeness, comparability and sensitivity. Data collected during these sampling events will be intended to achieve the following:

- Determine the seasonal and temporal metals loading in the Cement Creek watershed and Animas River.
- Determine the concentrations of metals in the Animas River water immediately, before during and after high flow conditions
- Determine the nature and extent of sediment metal contamination in the Animas River between Howardsville and Bakers Bridge
- Determine if there are additional sources of metals in the Animas River Canyon
- Determine the concentration of contaminants of COPECs in fish and invertebrate tissues to refine risk estimates of food chain exposures to wildlife receptors.

A.7.1 Planning Team and Stakeholders

The following sections list the members of the DQO planning team, primary decision makers, and parties who may be impacted by the results of this study or who may use this data generated as a result of the DQO process.

A.7.1.1 DQO Planning Team

The following table includes the DQO planning team members, respective organizations, and affiliation with that organization.

Table A.7-1 DQO Planning Team

Name	Organization	Area of Technical Expertise
Paula Schmittdiel	EPA Region 8	Remedial Project Manager
Dan Wall	EPA Region 8	Task Order Project Officer
Steven Way	EPA Region 8	On-Scene Coordinator
Sherry Skipper	USFWS	Biologist
Steve Auer	ESAT	Biologist
William Simon/Peter Butler	ARSG	Project Manager

A.7.1.2 Decision-Making Authority

The decision-maker has the ultimate authority for making final decisions based on the recommendations of the DQO team. The decision-maker for this event is Paula Schmittdiel, the EPA Region 8 Remedial Project Manager (RPM) for this site.

A.7.1.3 Stakeholders

Stakeholders are parties who may be affected by the results of the study or persons who may later use the data resulting from the DQO process. Table A.7-2 lists the impacted organizations and stakeholders, and the individuals that are representing those organizations.

Table A.7-2 Stakeholders

Organization	Represented By
EPA Region 8	Paula Schmittdiel
EPA Region 8	Steve Way
ARSG	Peter Butler/William Simon
USGS	Rob Runkel
Gold King Mines Corp	Todd Hennis
BLM	Kay Zillich
CDPHE	Craig Gander
Colorado Division of Reclamation	Kirstin Brown
Sunnyside Gold Company	Larry Perino
U.S. Fish and Wildlife Service	Sherry Skipper and Robyn Blackburn

A.7.2 Data Quality Objectives

The DQO process specifies project decisions, the data quality required to support those decisions, specific data types needed, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures that the resources required to generate the data are justified. The DQO process consists of the following seven steps:

1. State the problem,
2. Identify the goal of the study,
3. Identify the information inputs,
4. Define the boundaries of the study,
5. Develop the analytic approach,
6. Specify performance or acceptance criteria, and
7. Develop the plan for obtaining data.

During the first six steps of the process, the planning team develops decision performance criteria that will be used to develop the data collection design. The final step of the process involves developing the data collection design based on the DQOs. A brief discussion of these steps and their application to this project are provided in the following sections.

A.7.2.1 Step 1: State the Problem

Cement Creek, the receiving stream for the discharge of the American Tunnel, Red & Bonita, Mogul, and Gold King Level 7, is unable to support aquatic life. Cement Creek is a major contributor of metals and acidity to the Animas River. The river has goal-based cold water aquatic life standards. Presently fifteen total maximum daily loads (TMDL) allocations are not being met for Cement Creek and the Animas River.

Additional data are needed to better understand the potential risk to ecological receptors as identified in previous investigations. These data gaps were to be addressed during the fall of 2013 but due to unforeseen circumstances the sampling event was cancelled. The current state of contamination and its effect on aquatic life is still not understood to the extent that is needed for risk determination. Additional sampling is needed to identify possible sources of contaminants in the Animas River. In order to address these data gaps, further sediment, surface water, and macroinvertebrate samples need to be collected for laboratory analysis. This SAP will provide a plan for collecting these samples to address the data gaps and generate datasets to assist in further evaluation of site specific risks to aquatic life and wildlife receptors through exposure to biotic and abiotic media in the Animas River.

A.7.2.2 Step 2: Identify the Goals of the Study

The purpose of this step is to define the principle study questions (PSQs) that this study will attempt to resolve. The PSQs will help determine appropriate data inputs and potential alternative actions. Principle study questions can be used to develop decision statements when the potential alternative actions have been determined to resolve the problem. In situations where the outcomes may not lead to specific decisions or the information may be used to gain a greater understanding of existing data, estimation statements are more appropriate. Estimation statements are more applicable to the nature of the principle study questions (PSQs) being investigated in these sampling efforts.

The PSQ's are as follows:

PSQ1 – What is the seasonal and temporal variability of water chemistry and metals loads in Cement Creek and the Animas River?

PSQ2 – What are the concentrations of metals in the Animas River water immediately before, during and after high flow conditions?

PSQ3 – What is the nature and extent of metal contamination in the Animas River sediments?

PSQ4 – What are the metals concentrations in fish and invertebrate tissues in the Animas River?

PSQ5 – Are there additional sources of metals potentially impacting the Animas River in the Animas River Canyon?

Estimation Statements

PSQ 1: What is the seasonal and temporal variability of water chemistry and metals loads, in Cement Creek and the Animas River?

Results will be compared to those of previous sampling events to examine seasonal and temporal variability in water chemistry and metals loads in Cement Creek watershed and the Animas River.

PSQ 2: What are the concentrations of metals in the Animas River water immediately before, during and after high flow conditions?

High resolution surface water sampling for metals (MiniSippers) in the Animas River will be used to refine risk estimates to aquatic receptors during a period of sensitive life stages and high metals concentrations. (See Section B.2.2 for further discussion of the MiniSipper samplers).

PSQ 3: What is the nature and extent of metal contamination in the Animas River sediments?

Results from sediment and sediment pore water samples will be used to bolster existing data to better quantify the metals concentrations in sediment and associated pore water and refine risk estimates to benthic invertebrates in the Animas River.

PSQ 4: What are the metals concentrations in fish and invertebrate tissues in the Animas River?

Fish and macroinvertebrate samples will be collected and analyzed for metals to support estimating exposure of wildlife receptors to contaminants in food items.

PSQ 5: Are there additional sources of metals potentially impacting receptors the Animas River in the Animas River Canyon*?*

Specific conductance loggers will be deployed to bracket drainages in the Animas River canyon between Elk and Cascade Creeks immediately before, during and after runoff. Stations with significant deviations from other stations will be evaluated to help isolate potential sources of contaminations. Additional investigation may be warranted to determine the nature and cause of variations in specific conductance.

A.7.2.3 Step 3: Identify Information Inputs

The purpose of this step is to identify the data required to answer the PSQs listed in section A.7.2.2. The primary information and decision inputs will be data generated from field instruments and laboratory analyses (analytical and biological), as well as established water quality for comparison.

Field parameters and non-sampling objectives include:

- Taking temperature, pH, specific conductivity, DO, and flow measurements or estimates
- Determining metals concentrations in water collected using a combination of grab, integrated and MiniSipper sampling methods
- Determining metals concentrations in sediments collected at Animas River sampling locations.
- Determining specific conductance measurements in the Animas River above and below selected drainages in the Animas River canyon
- Collecting benthic macroinvertebrates in the Animas River
- Surveying fish populations and collecting fish tissue
- Collecting pore water at select Animas River locations
- Photographing the sampling locations and other notable observations
- Geospatial location data using GPS

Analytical laboratory parameters for routine and opportunistic surface water samples will include:

- Total and dissolved TAL metals and hardness (calculated)
- Analysis of alkalinity and anions for all surface water samples
- DOC for all Animas River stations and Chicago Basin opportunistic sampling

Laboratory method detection limits will be below selected standards and applicable benchmarks. Macroinvertebrate and fish samples will be collected for demographics and metals analysis of tissues. Fish data collection will be conducted by Colorado Parks and Wildlife according to their protocols and sampling requirements.

The following factors will be evaluated in the overall decision-making process:

- Comparison of surface water and pore water results to applicable standards/benchmarks and water quality criteria according to Colorado Table Value Standards or National Recommended Ambient Water Quality Criteria
- Sediment analytical results will be compared to probable and threshold effect concentrations as described in *Development and Evaluation of Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems* (MacDonald et al., 2000).
- Comparison of analytical data to applicable historical data to evaluate changes in water chemistry and metals loading over time
- Macroinvertebrate and fish tissue samples will be used in food chain modeling for determining the level of exposure for wildlife receptors to site-specific chemicals
- Where feasible fish and macroinvertebrate demographics results will be compared to historical data and Colorado Multi-Metric Index (MMI).

A.7.2.4 Step 4: Define the Boundaries to the Study

The objective of this step is to define the spatial and temporal components of the study area. The scale of the decision making for the decision statement (DS) is defined by combining the population of interest with the spatial and temporal boundaries. Practical constraints that could interfere with sampling are also identified. Implementing this step helps ensure that the data are representative of the population.

A.7.2.4.1 Spatial Boundaries

The study area boundary comprises the Cement Creek watershed and Animas River from Bakers Bridge to Howardsville. Sources include mining-impacted discharges from inactive or abandoned mines within approximately two miles of the historic Gladstone town site. These are the Gold King 7 Level, the American Tunnel, and the Red & Bonita, Mogul, and Grand Mogul mines. If seeps or springs are identified that contribute to flows into and out of the Cement Creek drainage, they may also be sampled. Opportunistic water and sediment samples will be taken in the Chicago Basin along Needle Creek (and possibly other water sources) to the confluence with the Animas River.

A.7.2.4.2 Temporal Boundaries

Sampling activities will start in April 2014 and end in September 2014. Specific dates are dependent on river flow conditions and availability of transportation to Animas River sample locations. The current schedule is as follows:

- April 14-15, 2014 — MiniSipper and STILs Deployment with Surface Water and Sediment Collection
- May 5-9, 2014 — High flow Sampling Event
- July 15-16, 2014 — MiniSipper and STILS Extraction
- September 22-26, 2014 — Low flow and Biota Sampling Event

A.7.2.5 Step 5: Develop the Analytic Approach

Total recoverable metals results and stream discharge measurements will be used to calculate loading of site related contaminants from contributing sources. The results from each of the inputs will be summed to determine the total loading of site related contaminants in Cement Creek into the Upper Animas River. Concentrations of dissolved metals in surface water and pore water will be compared to applicable standards, benchmarks, and water quality criteria either with the Colorado Table Value Standards or National Recommended Ambient Water Quality Criteria. Concentrations of metals in sediments will be compared to Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems (MacDonald et al, 2000). All analytical results will have detection limits suitable for comparison to these benchmarks and standards. Macroinvertebrate and fish tissue results will be used in food chain modeling for wildlife receptors. Where feasible, fish and macroinvertebrate demographics will be compared to historical data and Colorado Multi Metric Index.

A.7.2.6 Step 6: Specify Performance or Acceptance Criteria

The purpose of this step is to specify the tolerable limits on decision errors, which are used to establish performance goals for the data collection design. For this project, the number of samples and station locations are based largely on past investigations performed in 2009, 2010, 2011, 2012, and 2013 and the data collected will build upon previously collected data in order to expand the understanding of the site.

In order to mitigate the potential for false positive or false negative errors associated with field sampling, sample collection processes will be consistent with established and relevant SOPs. This includes collection of duplicate samples (and subsequent analysis using relative percent difference (RPD) statistics), implementing a decontamination procedure (which may include the use of disposable sampling equipment), and the use of field blanks. For laboratory analysis of samples, QA/QC steps (such as the use of laboratory controls, matrix spikes, matrix spike duplicates, blanks, etc.) will be consistent with ESAT Region 8 reporting requirements.

A.7.2.7 Step 7 Develop Plan for Collecting Data

A judgmental sampling design as described in *Guidance for Choosing a Sampling Design for Environmental Data Collection,* December 2002 (EPA QA/G-5S) will be used to assist with identification and verification of the sources of COPECs. Sampling locations were identified from the historical sampling locations and stations were refined in 2014 to bracket mine influences from the Gold King 7 Level, the American Tunnel, the Red & Bonita, Mogul, and Grand Mogul mine sites as well as any tributaries downstream of A72 and upstream of A72, Howardsville (A55) that may be a source of COPECs. Data collected from this event will assist with identifying the order and magnitude of contamination reaching the Upper Animas River and extent of migration to downstream locations through Bakers Bridge. Specific parameters of interest for the sampling events will follow the criteria listed in Section A.7.2.2 and are describe in Tables A.7-5 through A.7-7. Analytical methods for the events are described in Section B.4 and management of the data is described in B.10 of this document.

A.7.2.8 Sampling Locations

The specific parameters of interest and station locations were based on results from the previous sampling events that took place during 2009 through 2013. Sampling locations, descriptions, and activities that will take place in 2014 are listed in Tables A.7-5 through A.7-7 and shown in Figures A.7-1 through A.7-3. Table A.7-8 shows the GPS coordinates for each of the sampling locations. Sampling locations will be verified to the sub meter using Trimble GPS hand held devices as needed.

A brief description of the sampling locations will be recorded in the site-dedicated field notebook for each site sampled. Information will consist of sampling location identification number, date, time, access information, geographical observations, and any other pertinent information that will be useful in identifying the sampling location in the future. A photolog will be kept for locations and activities and photo identifications as assigned by the camera will be logged in the site-dedicated field notebook. If there are deviations from this SAP/QAPP or applicable SOPs including the decision to not sample a location because conditions are either unsafe or there are accessibility problems, these changes will be recorded in the site-dedicated notebook.

A.7.3 Criteria, Action Limits, and Laboratory Detection Limits

Tables A. 7-3 and A.7-4 provide the method detection limits (MDLs) and practical quantitation limits (PQLs). In every case, the MDLs and PQLs fall well below the screening criteria, indicating that the analytical methods will be able to measure contaminant levels in the surface water samples with the required sensitivity.

A.7.4 Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity

The documentation of the data evaluation effort will be in the form of the worksheets prepared during validation. These worksheets will be an appendix to the Sampling Analysis Report (SAR). The SAR will be prepared to identify problems that may affect data usability or require that the data be qualified. The SAR report will discuss all precision, accuracy, representativeness, completeness, comparability, and sensitivity parameter results from the data validation and overall usability of the data for project objectives, include the following:

Precision:
- Field duplicates: RPD criteria met?
- Laboratory duplicates: RPD criteria met?
- Method of standard dilution performed and criteria met?
- Matrix spike duplicates (MSD): RPD criteria met? (If applicable)

Accuracy:
- Matrix spike (MS/MSDs): (percent recovery) %R criteria met?
- Laboratory control sample/laboratory control sample duplicates: %R criteria met?
- Initial and continuing calibration recoveries met?
- Interference check sample recoveries met?
- Inductively coupled plasma serial dilution recoveries met?

Representativeness:
- Sampling procedures and design: criteria met?
- Holding times and preservation: criteria met?
- Custody: all chain-of-custody forms complete and provided in data package?
- Blanks: contaminants present?

Completeness:
- The number of valid analytical results are comparable (90%) with the number determined necessary during establishment of DQOs.

Comparability:
- Data compares with similar analysis and data sets?
- Sample collection methods comparable to similar data sets?
- Laboratory analytical methods comparable to similar data sets?

Sensitivity:
Method reporting limits met project objectives?

The data will be assessed for the following criteria:

- *Bias* – a systematic or persistent distortion of a measurement process that causes errors in one direction. The extent of bias will be determined by evaluating the laboratory initial calibration/continuing calibration verification, laboratory control spike/laboratory control spike duplicates (LCS/LCSD), blank spikes, MS/MSD, and method blanks.
- *Sensitivity* – the ability to discriminate between small differences in analyte concentration is related to the rate of change in response when there is a small change in stimulus and is reflected in the calibration curve. The detection limits of the field and laboratory methods are within the range of previous detections found at the site.
- *Precision* – the measure of agreement among repeated measurements of the same property under identical, or substantially similar, conditions and which is expressed as the RPD between the sample pairs. An acceptable RPD for water samples is 20% and 35% for soil and sediment (EPA, 2010)
- *Representativeness* – the measure of the degree to which data accurately and precisely represent a characteristic of a population parameter, variations at a sampling point, a process condition, or an environmental condition.
- *Completeness* – a measure of the amount of valid data obtained from a measurement system. The actual percentage of completeness is less important than the effect of completeness on the data set. Completeness will be assessed by the total number of samples collected versus the amount of samples planned.
- *Comparability* – the qualitative term that expresses the confidence that two data sets can contribute to common interpretation and analysis; comparability is used to describe how well samples within a data set, as well as two independent data sets, are interchangeable.

Uncertainty of validated data will be evaluated by the RPM or his designee to determine if the DQOs were met. In the event that the DQOs were not met, they will be reviewed to determine if they are achievable and may be revised if necessary, and the data may be further evaluated to determine the impact to the project. Data usability and limitations will be evaluated by the RPM.

A.8 Special Training/Certifications

All field staff have completed the Occupational Safety and Health Administration (OSHA) 40-hour Health and Safety Course for Hazardous Waste Site Worker Training in accordance with Sections e and p of OSHA 29 Code of Federal Regulation (CFR) 1910.120 and maintain this certification with annual eight-hour Hazardous Waste Site Operations Refresher Training as required by Sections e and q of OSHA 29 CFR

1910.120. Field staff has completed American Red Cross Standard First Aid and Adult CPR Training and maintain this certification annually for Adult CPR and every two years for Standard First Aid. The ESAT and EPA Health and Safety Managers are responsible for ensuring that all field staff completes the training requirements as required by OSHA.

A.9 Documentation and Records

Field measurements will be recorded in a site-dedicated field notebook at the time of data collection. A brief description of the stream flow measurements will also be recorded in the field notebook. Flow measurement data will be stored in the individual Flow Tracker unit and downloaded as soon as practical. The data sheets from the data download will be printed; scanned copies will be included in the sampling activities report. Field notebooks, chain-of-custody forms, groundwater sampling forms, and other forms used for the site investigation will be stored at the Region 8 EPA Laboratory Suite A127.

The documentation of the data evaluation efforts will be in the form of the worksheets prepared during validation. These worksheets will be an appendix to this SAR. The SAR will be prepared to identify problems that may affect data usability or require that the data be qualified. The SAR will discuss all precision, accuracy, representativeness, completeness, comparability, and sensitivity parameter results from the data validation and overall usability of the data for project objectives.

The laboratory will submit to EPA a data report containing all the analytical results for this sampling effort. The report will contain a case narrative that briefly describes the number of samples, analyses, and any analytical difficulties or QA/QC issues associated with the samples. The data report will also include signed chain-of-custody forms, analytical data, a QA/QC package, and raw data. Additional reporting requirements are outlined in the ESAT laboratory contract and quality management plan (QMP).

Peer review of the data package, at a 100% frequency of reported versus raw data, will be performed by the analytical laboratory. The final report of the abbreviated data validation will be in a standard Contract Laboratory Program (CLP) format, including all laboratory and instrument QC results.

B. DATA GENERATION AND ACQUISITION

This section describes data generation and acquisition activities associated with these events, including process design, sampling and analytical methods, sample handling and custody, QC, equipment, and data use and management.

B.1 Sampling Process Design

The following sections describe the sampling methods to collect and analyze surface water, sediment, macroinvertebrate, and fish tissue samples. Appendix A provides copies of the applicable SOPs, outlining how field activities will be performed (including documentation protocols). Tables B.1-1 and B.1-2 provide the sampling checklist and field equipment checklist, respectively. Section A.8 provides the rationale for the sampling process outlined in this section.

Sampling will occur from most downstream to most upstream sampling locations to avoid cross-contamination in accordance with SOP# FLD-1 *Surface Water Sampling* (ESAT, 2012). In addition, care will be taken at each sampling location to first collect the surface water samples before collecting the sediment samples in order to minimize the chances of contaminating the surface water with sediment particles.

The EPA RPM, or her designee, will be responsible for directing corrective actions if problems are encountered in the field which would impact the way this SAP/QAPP is implemented, or if sampling locations are inaccessible. Dan Wall is the EPA designee appointed for the studies. Any problems encountered and actions taken or deviations from this SAP/QAPP will be documented in the field notebook.

B.1.1 Surface Water Sampling

Surface water samples will be collected from April to July 2014 at seven locations using an automated sampler, the MiniSipper (Table A.7-5). Conductivity will be measured from April to July 2014 using the STIL and data downloaded from the unit after collection in July. Water grab samples will be collected at the MiniSipper locations upon deployment and periodically (up to once a week) at a MiniSipper location near the town of Silverton for QA purposes. High and low flow samples at locations on Cement Creek and the Animas River will be collected during May and September (Tables A.7-6 and A.7-7). Opportunistic samples in the Chicago Basin area will be collected in late July. Tables A.7-5 through A.7-7 provides the names and a description of each of the surface water sampling locations as well as the sampling activities scheduled to occur.

B.1.2 Pore Water Sampling

Pore water will be collected during the April and September events for use in evaluating potential ecological impacts to the existing contaminants in the hyporheic zone of the streambed. Water samples will be analyzed for dissolved metals. Analytical methods and detection limits are shown in Table A.7-3. Pore water samples will be collected using a Push Point sampling device with a syringe. Specific procedures for using the Push Point sampler are included in *Pore Water Sampling* SOP# FLD-10 (ESAT, 2012).

B.1.3 Sediment Sampling

Sediment samples will be collected during the April and September sampling events at the locations listed in Tables A.7-5 and A.7-7 for determination of contaminant loading in streambed sediments. Samples will be analyzed for total recoverable metals and mercury. Analytical methods and detection limits are shown in Table A.7-4. Sediment samples will be collected using Teflon scoops in accordance with the protocols outlined in *Standard Operating Procedures for Shallow Stream Sediment Sampling,* FLD-06 (ESAT, 2012)

B.1.4 Macroinvertebrate Sampling

Macroinvertebrate samples will be collected to further understand the demographics of the population and to measure the levels of COPECs in each sample (as a composite sample per location). The method detection limit MDL for these samples is dependent on the mass of each composited sample and cannot be determined until samples are processed in the laboratory. Every effort will be made in the field to collect sufficient samples in order to achieve the detection limits as shown in Table A.7-4 for total recoverable metals. The minimum mass of the samples needs to be 1.0 g dry weight and personnel involved with collection of invertebrates should fill the supplied DigiTube ™ sample containers approximately one half to three quarters full in accordance with Rapid Bioassessment Protocols methods.

B.1.5 Fish Sampling

Fish population surveys will be conducted by Colorado Parks and Wildlife in order to evaluate the diversity and population of fishes in the Animas River. Five individuals of each species of fish collected at a sampling site will be euthanized and tissue will be analyzed for determination of COPEC levels. Data from the tissue analysis will be used in food chain modeling. Fish survey results will provide information on fish species diversity, population structure, reproductive success and body condition of fish populations found in the Animas River. The t pass removal estimator (Bagenal, 1978) will be used to estimate the population. Detection limits for tissue analysis will follow those shown in Table B.5-1 for total recoverable metals.

B.1.6 Nature of Data Collected

As indicated in Section A.7, a variety of data will be collected during these events, some of which are critical to achieve the established DQOs and project objectives, and some of which are primarily for informational purposes or which will be used supplement critical data. The following chart specifies each type:

Data Type	Purpose
Real-time water quality parameters (pH, temperature, conductivity and DO)	Critical
Stream flow measurements	Critical

Surface water (analyzed for dissolved metals and hardness, total recoverable metals, DOC, anions, and alkalinity)	Critical
Sediment (analyzed for total recoverable metals)	Critical
Macroinvertebrates	Critical
Fish	Critical
GPS coordinates	Critical
Photolog	Informational
General field observations noted in logbook	Informational

B.1.7 Data Variability

As indicated in Section A.7, a variety of data will be collected during these events, some of which are critical to achieve the established DQOs and project objectives, and some of which are primarily for informational purposes or which will be used to supplement critical data.

B.2 Sampling Methods

This section describes surface water, pore water, sediment, macronivertebrate and fish sampling methods that will be employed during these sampling events as well as applicable SOPs, necessary equipment and support facilities.

B.2.1 Equipment and Support Facilities

Specific field equipment necessary for execution of the SAP are included in Table B.1-2. During field activities, additional support facilities and vehicles outside of the sampling vehicle are not anticipated to be needed.

B.2.2 Sampling for Surface Water

Sampling will progress from downstream to upstream locations to eliminate the potential for sediment disturbance in the stream bed that could result in cross contamination of subsequent samples. Surface water samples will be collected by immersing the sample bottle several inches beneath the water surface with the mouth of the sample bottle facing upstream. To collect such a sample, the sample container will be inverted, lowered to the approximate sample depth and held approximately at a 45-degree angle with the mouth of the bottle facing downstream. The bottle will be rinsed three times with stream water from the sample location prior to collecting the sample.

If surface water samples cannot be collected directly into the sample container, a decontaminated 1-liter bottle or cubitainer will be used to collect the sample. The bottle will be rinsed three times with water from the sample location prior to collecting the sample. Care will be taken to avoid excessive agitation when transferring samples to the sample containers.

For water samples in the Animas River, equal discharge increment technique (EDI) will occur during the low flow event and will require protocols specified in the "*Operators Manual for the US DH-81 Depth-Integrated Suspended-Sediment Sampler*" (FISP, No date). This technique will be employed during the low flow event at all locations warranted for the technique to be used. It will be at the discretion of the field team lead to determine if the sampling location is suitable to perform this type of sampling using the DH-81. This process of using the EDI technique will provide an equal volume of sample at each stream vertical from the cross section of the stream or river. Attachment A shows the filling time for the US DH-81 sampler using a one liter bottle and this is used to calculate the transit rate. The transit rate is determined by multiplying the steam depth at the sampling vertical by 2 and divide by the sampling time after determining the velocity of the stream. The result is the rate in ft/sec (FISP, No date). Table B.2-1 shows the filling time for the DH-81 sampler using a 1one liter bottle.

Also, seven locations have been selected for MiniSipper sampler deployment (See Figure A.7-3). As described by Chapin and Todd (2012) the MiniSipper consists of the following components:

- Waterproof housing containing a microcontroller, three solenoid micro-pumps and a rechargeable battery
- Sample coil with approximately 150 meters of 3.2 millimeter outer diameter by 1.6 millimeter inner diameter high purity perfluoroalkoxy tubing
- Collapsible bag for a nitrogen gas bubble separator
- 50 millimeter coil of nitric acid stabilizing reagent

The MiniSipper injects 5 milliliter water samples into the sample coil. Water samples are then preserved with 0.25 milliliters of nitric acid (stabilizing reagent) to a pH of 2 and separated via injection of a nitrogen gas bubble into the sample line. Samples are filtered in situ with a 10 micron ultra-high molecular weight polyethylene solvent filter. Approximately 250 samples can be loaded into the sample coil.

B.2.3 Sampling for Pore Water

Pore Water samples will be collected by inserting a PushPoint® sampler into the hyporheic zone and purging until the pore water runs clear in accordance with *Pore Water Sampling*, SOP#FLD-10 (ESAT, 2012). The syringe used to extract the pore water will be rinsed three times prior to sample collection with water from the Animas River. The water samples pulled into the syringe will be placed in a 250 mL High Density Polyethylene (HDPE) sample bottle. After approximately 250 mL of water has been collected the sample will be filtered by using a 0.45 micron filter. The push point pore water samplers will be decontaminated between uses in accordance with *Sampling Equipment Decontamination* SOP# FLD-2 (ESAT, 2012).

B.2.4 Sampling for Sediment

Sediment samples will be collected using dedicated Teflon scoops and placed in HDPE containers based on the protocols outlined in *Shallow Stream Sediment Sampling* SOP# FLD-6 (ESAT, 2012). Sampling locations that have not been previously recorded will be documented following *Global Positioning System (GPS) – Trimble GeoXT 2008 Series,* SOP# FLD-7 (ESAT, 2012) (Appendix A) and given an appropriate sample designation that is consistent with sampling location nomenclature for the site.

Several sediment samples will be collected from a stretch of creek 50 meters upstream to 50 meters downstream of the actual sampling location. These subsamples will then be combined and homogenized back at the laboratory. The goal is to obtain up to 60 mL of representative sediment from each sampling location and the required sample volume needed for the analyses of the sediment samples, which consists of 200 g for total recoverable metals and total mercury analysis. No sampling equipment needs to be decontaminated since the Teflon scoops are not re-used across sampling locations.

B.2.5 Sampling for Macroinvertebrate and Fish

Macroinvertebrate samples will be collected using a semi-quantitative approach during the September 2014 sampling event at the locations identified in Table A.7-7. Macroinvertebrate sampling will be performed using a D-frame dip net (one foot base length). Samples will be collected from a 100 meter stretch (representative of the characteristics of the stream) at each sampling location. Samples will be collected by inserting the D-frame dip net into each identifiable stream characteristic (riffle, run, and pool) and disturbing about one square foot of substrate (using the toe or heel of a boot) upstream from the net for a one minute time period in accordance with Rapid Bioassessment Protocols methods. Large substrate particles may need to be picked up and rubbed by hand to dislodge attached organisms. After each one minute interval, wash the debris and organisms from the net into a sieve using stream water, transferred to the sample container, and then preserved with ethanol. This process will be repeated two more times in each attribute (for a total of three replicate samples, each of which are a composite of unique run, riffle, and pool samples).

Fish population surveys will be conducted by Colorado Parks and Wildlife during the September event using the two pass removal estimator technique (Bagenal, 1978). A tote barge electrofisher will be used to stun fish so they can be easily collected. Electro fishing will commence downstream to upstream. Two team members will each hold an anode pole and be responsible for netting the specimens while a third is responsible for pushing the barge. Once netted, the fish are placed in the live well on the barge which is outside the area of electric current. All fish over 20 millimeters in length will be identified down to the species and examined for external anomalies before being released or collected for tissue analysis. It is anticipated that five of each species will be collected during these efforts.

B.3 Sampling Handling and Custody

Sample designation will consist of a series of letters and numbers to indicate site and location. Tables A.7-5 through A.7.7 show the naming convention used and the site descriptions. The sampling areas are as follows:

- A Animas River
- ATS American Tunnel Seep
- Bbridge Bakers Bridge
- FD Fenn Drainage
- CC Cement Creek
- M Mineral Creek
- MTD Mogul Tailings Drainage

All samples will be preserved and transported back to the EPA Region 8 laboratory following chain-of-custody protocols. The samples will be relinquished to the sample custodian at the laboratory, together with the completed and signed chain-of-custody forms. **Attachment A** provides a blank chain-of-custody form. The sample custodian will inspect the coolers upon arrival to make sure that the proper temperature was maintained, that the sample containers are intact and sealed, and that the number of samples in the coolers match the information provided in the chain-of-custody forms. All the samples will be stored in an access-controlled sample cooler at the laboratory. An analytical chemist will log the samples in the Laboratory Information Management System (LIMS) upon receipt and will enter all analytical data into the SCRIBE database for permanent storage/archiving.

All field measurements and observations will be recorded in a bound notebook or on appropriate data sheets by the field personnel at the time they are performed in accordance with the Contract Laboratory Program Guidance for Field Samplers (EPA, 2011b). The personnel doing the recording will initial and date all measurements, observations, and any other notations made. Corrections will be performed by drawing a single line through the error accompanied by the date and the initials of the person performing the correction, followed by the proper entry. Chain-of-custody forms will be filled out during the time of collection and will follow protocol provided in *Sample Custody and Labeling,* SOP# FLD-11 (ESAT, 2012).

B.3.1 Surface Water Sample Preservation

Surface water samples collected for analysis of total recoverable metals and dissolved metals will be acidified in the field using ultra-pure nitric acid to a pH < 2. Alkalinity and anions samples will not be chemically preserved. Samples will be stored in coolers on ice and kept at approximately 4℃ for transport to the EPA Region 8 Laboratory in accordance with *Standard Operating Procedure for General Field Sampling Protocols* FLD-12 (ESAT, 2011b). Samples collected by the MiniSipper will be filtered using a ten micron filter and preserved in-line prior to the inert gas separator. MiniSippers will be deployed for approximately 13 weeks.

The maximum holdings times differ depending on the target analytes or compound, as follows (see also Table A.7-3): The holding times are 180 days for total recoverable metals and dissolved metals, 28 days for DOC and anions, and 14 days for alkalinity.

B.3.2 Pore Water Sample Preservation

Pore water samples will be filtered and then acidified in the field using ultra-pure nitric acid to a pH< 2. Samples will be stored in coolers on ice and kept at approximately 4℃ for transport to the EPA Region 8 Laboratory in accordance with *Standard Operating Procedure for General Field Sampling Protocols* FLD-12 (ESAT, 2011b). The maximum holdings times differ depending on the target analytes (see also Table A.7-4). The holding times are 180 days for dissolved metals.

B.3.3 Sediment Sample Preservation

The sediment samples will be immediately stored in coolers on ice and kept at approximately 4℃ and then transported to the EPA Region 8 Laboratory in accordance with *General Field Sampling Protocols* SOP# FLD-12 (ESAT, 2012). The maximum holdings times differ depending on the target analytes (see also Table A.7-4). The holding times are 180 days for all metals, except for mercury which has a holding time of 28 days.

B.3.4 Macroinvertebrate and Fish Sample Preservation

Macroinvertebrates collected for demographics will be preserved using 90% ethanol and it is important that the one liter HDPE container is filled no more than 50% of stream material (including all sticks, pebbles, sand, and invertebrates contained within). These samples will be placed in a cooler during transport and shipment to the analytical laboratory. Macroinvertebrate samples collected for tissue analysis will be placed in a HDPE wide mouth sample bottle and placed on ice during transport. Samples will be frozen at the Region 8 Laboratory until chemical analysis is performed. Holding time for these samples is 180 days for all metals with the exception of mercury which is 28 days.

Tissue samples from fish will be placed in plastic bags and on ice after collection and during transportation to the Region 8 Laboratory. Samples will be frozen at the Region 8 Laboratory during the holding time of 180 days for metals and 28 days for mercury.

B.4 Analytical Methods

Samples will be analyzed for total recoverable metals, dissolved metals, DOC, alkalinity, and anions. Tables A.7-3 and A.7-4 include the laboratory analytical instrumentation and methods to be used for sample analysis. These methods will be in accordance with EPA *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, also known as SW-846, Method 7473, Revision 0, January 1998. Additionally, sample analysis will be in accordance with Method 200.7 *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*, Revision 4.4, May 1994, and Method 200.8 *Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry*, Revision 5.4, May 1994 and Method 245.1, Revision 3.0 *Determination of Mercury in Water by Cold Vapor Atomic Absoption Spectrometry*.

Sample disposal of potentially hazardous waste will follow protocol defined in *Collection, Analysis and Disposal of ESAT Laboratory Waste* SOP LAB01.01 (ESAT, 2012).

Field parameters measured at each sample site will include dissolved oxygen, temperature, pH, conductivity, and stream flow. Physical observations of the stream will be noted in the field notebook. Photographs of the site will be taken and will be documented as an appendix to the 2014 SAR.

In the event that problems are encountered in the field that may impact the implementation of this SAP/QAPP, the EPA RPM, or her designee will be responsible for directing corrective actions. The designee appointed for these events is Dan Wall. Any problems encountered and actions taken or deviations from this SAP will be documented in the field notebook and stakeholders will be notified of changes within 72 hours.
It is currently anticipated that ESAT will be used for sample analysis during this project. Laboratory instrumentation and method requirements for the ESAT laboratory are included in Tables A.7-3 and A.7-4.

B.4.1 Surface Water

Surface water samples collected will be submitted for analysis to the EPA Region 8 Laboratory, ESAT Analytical Chemistry department. Specific analysis for each event is presented in Tables A.7-5 through and A.7-7. Samples will be collected, preserved, labeled, and stored in accordance with the *Field Sampling Protocols,* SOP # FLD-12 (ESAT, 2012). The sample protocols for surface water are provided in Table B.5-1 and B.5-2.

B.4.2 Sediment

Sediment samples will be submitted for analysis to the ESAT analytical chemistry department at the EPA Region 8 Laboratory. Specific analysis for each event is presented in Tables A.7-5 through A.7-7. Samples will be collected, preserved, labeled, and stored in accordance with the *Field Sampling Protocols,* SOP # FLD-12 (ESAT, 2012). The sample protocols for sediment are provided in Table B.5-1.

B.4.3 Macroinvertebrate and Fish

Macroinvertebrate and whole fish tissue samples will be submitted for total recoverable metals analysis to the ESAT analytical chemistry department at the EPA Region 8 Laboratory. Specific analysis for each event is presented in Table A.7-7. Samples will be collected, preserved, labeled, and stored in accordance with the *Field Sampling Protocols,* SOP # FLD-12 (ESAT, 2012).

B.5 Quality Control

Tables B.5-1 and B.5-2 provide acceptable laboratory QC criteria for ESAT and EPA Region 8. The sample selection for laboratory QC will be determined by the laboratory staff. Where a specific QC criteria table is not provided, the method's QC requirements are met or exceeded by ESAT's and EPA's analytical process.

The calibration procedures for the field measurements to be performed using the In-Situ water quality meter are detailed in the *Water Quality Measurements with the In-Situ® Multi-Parameter Meter* SOP # FLD-09 (ESAT, 2012). If other multi-probes are used for this sampling event, the field sampling team will calibrate the probe according to the manufacturers specifications listed in the owner's manual. The SOPs and procedures appended to this document also detail the associated QA and/or QC criteria for the field analyses and equipment.

Field QC samples will be collected on the following basis:

- Filter/container/preservative blank – minimum of one blank per 20 samples collected or one field blank per day
- Duplicates (collocated) – minimum of one duplicate per ten samples collected
- QC samples will be collected weekly from each of the MiniSippers – minimum of one sample from each of the seven MiniSipper locations each week until the end of the project (15 weeks), for a total of 90 samples

B.6 Instrument/Equipment Testing, Inspection and Maintenance

The following chart includes the equipment that will be used during execution of this SAP that requires testing, inspection and/or maintenance.

Equipment/Instrument	Requirement	Schedule
MiniSipper	Calibration, routine maintenance, scheduled service	In accordance with manufacturer's specifications and user's manual
In-Situ® Multi-Parameter Meter	Calibration, routine maintenance, scheduled service	In accordance with manufacturer's specifications, user's manual and applicable SOPs
Trimble®GeoXT™GPS	Service	As needed depending on equipment performance
Sontek®Flowtracker™	Calibration, routine maintenance, scheduled service	In accordance with manufacturer's specifications, user's manual and applicable SOPs
Laboratory analytical instrumentation	Calibration, routine maintenance, scheduled service	In accordance with manufacturer's specifications, user's manual and applicable SOPs
Electro Fishing Equipment	Maintenance and service	In accordance with manufacturer's specifications and user's manual

Maintenance and servicing schedules as well as applicable testing criteria are included in the applicable user's manuals as well as SOPs (attached to this document). Note that most spare parts for each piece of equipment are kept at the Region 8 Laboratory, including parts for field equipment as well as laboratory instrumentation. Spare parts are routinely available and are ordered during periodic maintenance activities to ensure they are on hand when needed. Services agreements are in place for all laboratory instrumentation to address equipment maintenance, service, parts and repair needs as they arise. Equipment and instrument calibration requirements and frequencies are detailed in the applicable SOPs and user's manuals (attached to this document).

Field equipment will be inspected, tested and routine maintenance performed prior to deployment in the field by contractor staff members at the EPA Region 8 Laboratory knowledgeable of equipment operation and maintenance requirements. Any equipment deficiencies and maintenance requirements will be identified and mitigated (i.e., parts replaced, alternate equipment deployed, etc.). After mitigation, equipment will be re-inspected and the effectiveness of any repairs will be verified. Any repair or maintenance activities performed will be documented in the applicable equipment or instrument log book. Backup equipment will be deployed during these events in case of equipment or instrument failure in the field.

B.7 Instrument/Equipment Calibration and Frequency

As indicated in Section B.6, some laboratory instrumentation (analytical instrumentation) and field equipment (such as water quality meters and flow meters) will require periodic calibration to verify function. Calibration requirements, procedures, testing criteria and deficiency resolution procedures are included in applicable SOPs and user's manuals, each of which are included in Appendix A of this document (for field equipment). SOPs and user's manuals for laboratory analytical instrumentation are on file and readily available at the Region 8 Laboratory. Any variations or inability to calibrate a piece of equipment or instrument will be noted in the relevant logbook, and appropriate mitigation procedures will be followed, or replacement equipment will be obtained. Recalibration of any instrument that requires mitigation of a deficiency will be performed prior to use or deployment.

B.8 Inspection/Acceptance of Supplies and Consumables

All supplies for this event will be purchased by the EPA from approved vendors, and stored in the field sampling room (or adjacent storage rooms at the Region 8 Laboratory). The week prior to the sampling event, an EPA or ESAT sampling team member will gather needed supplies and consumables, which will subsequently be verified by an ESAT team member. Supplies and consumables will be ordered, inspected upon receipt, accepted, tracked, and inventoried by the EPA field biologist at the Region 8 Laboratory. Acceptance of supplies and consumables will be based on the requirements of the end user.

B.9 Use of Existing Data (Non-Direct Measurements)

Non-direct measurements were relied upon for preparation of project implementation. These measurements include previous sampling and analysis plans (EPA, 2013), historical data from ARSG, and past Sampling Activities Reports.

B.10 Data Management

Specific management processes will be followed for data likely to be collected during field activities: field equipment calibration and maintenance entries, field logbook entries, chain-of-custody forms, electronically entered and logged data (such as GPS locations, flow measurements, etc.), and analytical data.

Field equipment calibration and maintenance logs – All field equipment calibration and maintenance activities will be documented in a logbook dedicated to each piece of equipment. Logbook entries will be signed and dated by the individual performing calibration or maintenance, or the individual responsible for coordination (such as the field task lead) if equipment is shipped to a manufacturer for repair and/or maintenance.

Field logbooks will be stored with the appropriate piece of equipment. When new logbooks are needed, the former logbook will be stored at the Region 8 EPA Laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements.

Field logbook/datasheet entries – All field measurements and observations will be recorded in a bound notebook or on appropriate data sheets by the field personnel at the time they are performed. The personnel doing the recording will initial and date each logbook. Corrections to logbook entries will be made by drawing a single line through the error accompanied by the date and the initials of the person performing the correction, followed by the proper entry. Upon return to the Region 8 laboratory, all data hand entered into field notebooks and datasheets will be transferred to electronic spreadsheets (such as Microsoft® Excel) by ESAT contract staff to prepare for uploading to a SCRIBE project (see below). ESAT field personnel will perform a 100% verification of spreadsheet entries against hand-entered field logbook and datasheet entries before uploading to SCRIBE. Original field notebooks and data sheets will be stored at the Region 8 EPA Laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements. Non-SCRIBE electronic files generated as a part of this process (i.e., spreadsheets, photographs, scanned logbooks) will be stored on the ESAT Region 8 contractor G drive.

Chain-of-custody forms – When possible, chain-of-custody forms will be generated prior to field activities using SCRIBE and will be filled out when samples are collected following the protocol outlined in *Sample Custody and Labeling* SOP# FLD-11 (ESAT, 2012). Otherwise, blank chain-of-custody forms will be used to collect sample information during field activities. Information entered on the forms during investigation activities will be entered into SCRIBE after returning to the Region 8 Laboratory as a part of the SCRIBE upload process (see below). ESAT personnel will verify 100% of all the data entered into SCRIBE against the chain-of-custody forms completed in the field. Hard copies of these forms will be stored at the Region 8 Laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements.

Electronically entered or logged data – In some cases data may be recorded in the field directly on electronic field forms or using data loggers (such as GPS instrumentation or multi-probe data loggers). In these cases, upon return to the Region 8 Laboratory, all electronic data logs will be downloaded directly to a spreadsheet (or alternate electronic media depending on specific instrument software requirements), verified against any hand-written documentation (such as field logs and field data sheets) and processed into an electronic form that can be uploaded directly to SCRIBE. Similarly, electronic field forms will be processed to allow for upload to SCRIBE. Electronic field forms and/or data logs will be maintained on the ESAT Region 8 contractor G drive. In cases where information must be manually entered into SCRIBE, ESAT personnel will perform 100% verification between electronic documents and data logs and data manually entered into SCRIBE.

Analytical Data – An analytical chemist will log all the samples into LIMS upon receipt at the Region 8 Laboratory. All analytical results will be uploaded into the LIMS in accordance with SOP# LAB-05.02, *Sample Receipt, Custody, Storage and LIMS Entry of Samples* (ESAT, 2009). Peer review of the data package, at a 100% frequency of reported versus raw data, will be performed by the analytical laboratory before a final report is released. The final report will be in a standard CLP format, including all laboratory and instrument QC results. The laboratory Electronic Data Deliverable (EDD) will immediately be uploaded into a SCRIBE project for permanent electronic storage and archiving after the final report is generated. Hard copies of data reports (including bench sheets) will be stored at the Region 8 Laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements.

SCRIBE project generation – As indicated above, all data generated as a part of field investigation activities will be uploaded into a SCRIBE project (or update to a SCRIBE project) and subsequently published to Scribe.net in accordance with the *Data Management for Field Operations and Analytical Support,* SOP# 16-DAT-01.00 (ESAT, 2014). It is anticipated that more data may be collected in the field that supersedes existing or historical data that has already been published (such as GPS locations, etc.) for a specific site. Therefore, before data are published or updated to SCRIBE projects, ESAT personnel will perform a 100% verification of each SCRIBE project against data collected in the field (hand entered logbook data, electronic forms and data logs) prior to publishing the project on Scribe.net. Verified SCRIBE projects will be published within one week of delivery of analytical EDD when possible. The EPA project manager will be immediately notified and an alternate publication date will be established. In the event that conditions preclude publication within that time period, the Task Order Project Officer (TOPO) will be notified and a new publication date will be established.

C. ASSESSMENT AND OVERSIGHT

C.1 Assessment and Response Actions

C.1.1 Field Sampling Assessments

Assessment and oversight of field sampling activities and implementation of the SAP/QAPP will include the following:

- Oversight of field sampling activities
- Oversight of sample handling and chain of custody procedures

The following individuals or their designees are authorized to perform any of the assessments listed above:

- EPA TOPO – Nicole Plescia
- EPA RPM – Paula Schmittdiel

Assessment of field activities may occur at any time and without prior notice. Only authorized individuals may conduct the assessments and it is their role to issue any corrective action or response action to the situation. If minor problems are identified they will be addressed on site prior to resuming work. If more significant problems are identified then a stop work order can be issued by the TOPO until the project manager or designee can resolve the problem.

C.1.2 Laboratory Assessments

System assessments of the designated laboratory may be performed by ESAT. The quality assurance officer (QAO), or a designee, may perform a laboratory inspection.

Routine assessments will be conducted at least once a year, in accordance with ESAT's QMP. However, the frequency of the laboratory system assessments will also be based on the level of use and performance of individual designated laboratories. A member of the ESAT team will perform the assessment in accordance with the assessment checklist and TechLaw SOP 02-06-05. The checklist requires examining the laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc. Routine assessments will also be performed before a laboratory is added to the approved laboratory list. Should one-time specialty analysis be requested, the need for on-site assessments will be evaluated and discussed with EPA before an audit.

Performance assessments will require preparing blind QC samples and submitting them along with project samples to the laboratory for analysis. The analytical results of the QC sample analyses are evaluated by the QAO to ensure that the laboratory maintains acceptable QC performance. Performance assessments may be requested by ESAT or EPA. Performance Evaluation (PE) samples will be prepared by and obtained from vendors. The QAO will designate if a PE sample shall be submitted. PE samples should be submitted if a laboratory has not recently passed an outside PE sample or as requested by EPA.

Response Actions

Corrective Action (CA) may be required at two phases corresponding to the two activities of data generation: 1) field activities (data gathering phase); and 2) laboratory activities (data analysis phase). CAs required as a result of the data analysis phase are initiated by the TechLaw QAO when analytical data are found to be outside the limits of acceptability, as specified in the laboratory SOPs.

C.1.3 Field Corrective Actions

CA required as a result of the field data collection phase is initiated by the TechLaw field team leader and may result from log reports or field assessments. QC needs to be implemented both during the development of the SAP and during sampling activities to ensure that CAs will not be required. CAs are initiated by ESAT if weaknesses or problems are uncovered as a result of field activities. The CAs will depend on the nature or severity of the problem and the level at which the problem is detected, and may include, but shall not be limited to:

- Modifications to sampling procedures
- Recalibration (or replacement) of field instruments
- Additional training of field personnel
- Reassignment of staff personnel
- Re-sampling

C.2 Reports to Management

The results of all laboratory assessments will be submitted to the appropriate ESAT project manager, task order manager, and laboratory assistance team, as well as the EPA Contracting Officer Representative and EPA QA personnel, if requested. An external assessment of the designated laboratory may also be conducted by EPA, at the Region's discretion.

D. DATA VALIDATION AND USABILITY

D.1 Data Review, Verification, and Validation

Laboratory data validation and verification will begin at the sample log-in stage where a sample log-in technician or chemist will compare received samples against chain-of-custody forms and document sample condition (damage, temperature, etc.). Validation and verification of data will be performed by QA/QC personnel following EPA National Functional Guidance for Inorganic Data, (EPA, 2002) in order to determine if the DQOs were met. Sample data deemed outside the expected range will be investigated, communicated to the analytical chemistry staff, flagged (if needed) and potentially re-sampled to verify or discredit the data. Data that have proven to be incorrect may be flagged, further reviewed, or invalidated. The cause of incorrect data will be investigated and appropriate response actions will be taken, including communication of any issues to the user in the data report.

Uncertainty of validated data will be evaluated by the RPM to determine if the DQOs were met. In the event that the DQOs were not met, they will be reviewed to determine if they are achievable and may be revised if necessary, and the data may be further evaluated to determine the impact to the project. Data usability and limitations will be evaluated by the RPM.

Abbreviated verification will be completed on 10% of the analytical results for data that is electronically uploaded directly from the analytical instrumentation into the ESAT LIMS. This will be performed to ensure that data were produced in accordance with procedures outlined in this project plan. The following elements will be reviewed for compliance as part of the abbreviated data validation:

- Holding Times
- Calibration
- Blanks
- Spikes
- Duplicates
- LCSs
- Reporting Limits
- Analyte Quantification

D.2 Verification and Validation Methods

The analytical data will be validated for 10% of the results by either the acting EPA Region 8 Laboratory QA Officer or by a designated TechLaw, Inc. QA officer outside of the Region 8 ESAT office. The validation will include reviewing 10% of the samples for 100% of the analytical analysis performed and reported. The following elements will be reviewed for compliance as part of the abbreviated data validation:

1. Holding Times
2. Calibration
3. Blanks
4. Spikes
5. Duplicates
6. LCSs
7. MS/MSD
8. Post Digest Spike
9. Internal Control Standard
10. Dilution Sample
11. Reporting Limits
12. Analyte Identification
13. Analyte Quantification
14. Comparison of hardcopy results to EDD

Data validation will conform to the EPA Contract Laboratory Program National Functional Guidelines for Inorganic data and will use standard data qualifiers as described below.

D.3 Reconciliation with User Requirements

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process. If the regions choose to use additional qualifiers, a complete explanation of those qualifiers should accompany the data review.

U	The analyte was analyzed for, but was not detected above the level of the reported sample Quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the results may be biased high.
J-	The result is an estimated quantity, but the results may be biased low.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be presented in the sample.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise

D.4 Reconciliation with DQOs

Information obtained from the field investigation will be evaluated through the Data Quality Assessment (DQA) process to determine if the data obtained are of adequate quality and quantity to support their intended use. The DQA process consists of five steps, as summarized below (EPA, 2006):

1.) *Review the project's objectives and sampling design*: Review the objectives defined during the systematic planning to assure that they are still applicable. If objectives have not been deployed, specify them before evaluating the data for the projects objectives. Review the sampling design and data collection documentation for consistency with the project objectives observing any potential discrepancies.

2.) *Conduct a preliminary data review*: Review QA reports (when possible) for the validation of data, calculate basic statistics, and generate graphs of the data. Use this information to learn about the structures of the data and identify patterns, relationships, or potential anomalies.

3.) *Select the statistical method*: Select the appropriate procedures for summarizing and analyzing the data based on the review of the performance and acceptance criteria associated with the project objectives, the sampling design, and the preliminary data review. Identify the key underlying assumptions associated with the statistical tests.

4.) *Verify the assumptions of the statistical method*: Evaluate whether the underlying assumptions hold, or whether departures are acceptable, given the actual data and other information about the study.

5.) *Draw conclusion from the data*: Perform the calculations necessary to draw reasonable conclusions from the data. If the design is to be used again, evaluate the performance of the sampling design.

Uncertainty of validated data will be evaluated by the RPM to determine if the DQOs were met. In the event that the DQOs were not met, they will be reviewed to determine if they are achievable and may be revised if necessary, and the data may be further evaluated to determine the impact to the project. Data usability and limitations will be evaluated by the RPM.

References

Animas River Stakeholders Group (ARSG). 2001. Use Attainability Analysis for the Animas River Watershed. January 2001.

Bagenal, T. 1978. Methods for assessment of fish production in fresh water, 3rd edition. IBP Handbook #3, Blackwell Scientific Publications, Oxford, U.K.

Chapin, Thomas P. and Todd, A.S.. 2012. MiniSipper: A new in situ water sampler for high resolution, long duration acid mine drainage monitoring. Science of the Total Environment. 439:343-353.

CDPHE. Date Unknown. “Standard Operating Procedures for the Filtration of Water Samples.” From the Quality Assurance Project Plan for the Colorado Nonpoint Source Monitoring Program

MacDonald DD., Ingersoll CG., Berger TA. (2000) *Development and Evaluation of Consensus Based Sediment Quality Guidelines for freshwater Ecosystems*. Arch Environ. Contam. Tox. 39:20-31

Runkel, R.L., 2010, One-Dimensional Transport with Equilibrium Chemistry (OTEQ): A Reactive Transport Model for Streams and Rivers: U.S. Geological Survey Techniques and Methods Book 6, Chapter B6, p. 101 http://water.usgs.gov/software/OTEQ/

URS Operating Services (UOS). 2005. Gladstone Treatment Plant Targeted Brownfields Assessment Phase II Field Sampling Plan. July 2005.

URS Operating Services (UOS). 2006. Gladstone Treatment Plant Targeted Brownfields Assessment, Water Treatment Evaluation Report, September 14, 2006.

U.S. Environmental Protection Agency (EPA). 1991. Office of Emergency and Remedial Response, "Management of Investigation - Derived Wastes During Site Inspections OERR 9345.3-02."

U.S. Environmental Protection Agency (EPA). 1992. "Guidance for Performing Site Inspections Under CERCLA," Interim Final September 1992.

U.S. Environmental Protection Agency (EPA). 1993. "Region VIII Supplement to Guidance for Performing Site Inspections Under CERCLA." January 1993.

U.S Environmental Protection Agency (EPA). 1994. "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry", Method 200.7 Revision 4.4, May 1994.

U.S Environmental Protection Agency (EPA). 1994b "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry" Method 200.8 Revision 5.4, May 1994.

U.S. Environmental Protection Agency (EPA). 1998. "Quality Assurance Guidance for Conducting Brownfield Site Assessments." EPA 540-R-98-038 OSWER 9230.00-83P PB98-963307 September 1998.

U.S Environmental Protection Agency (EPA). 1998b EPA "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, Method 7473, Revision 0, January 1998

U.S. Environmental Protection Agency (EPA). 2000. "Guidance for the Data Quality Objectives Process." EPA QA/G-4. Copies may be obtained from the Quality Staff Home Page: www.epa.gov/quality August 2000.

U.S. Environmental Protection Agency (EPA). 2001a. "EPA Requirements for Quality Assurance Project Plans" EPA QA/R-5. March 2001.

U.S. Environmental Protection Agency (EPA). 2001b. EPA QA/G-5i 2001. Guidance on Data Quality

U.S Environmental Protection Agency (EPA). 2001c. "Requirements for Quality Assurance Project Plans" (EPA QA/R-5")

U.S. Environmental Protection Agency (EPA). 2002. Choosing a Sampling Design for Environmental Data Collection (EPA QA/G-5S). December 2002.

U.S Environmental Protection Agency (EPA). 2002. "Guidance for Quality Assurance Project Plans" (EPA QA/G-5).

U.S Environmental Protection Agency (EPA). 2006. "Guidance on Systematic Planning Using the Data Quality Objectives Process" *(EPA QA/G-4).*

Walton-Day, K., Runkel, R.L., and Kimball, B.A., in press, Using spatially detailed water-quality data and solute-transport modeling to support Total Maximum Daily Load development. Journal of the American Water Resources Association.

Standard Operating Procedures:

Environmental Services Assistance Team (2014) *Data Management for Field Operations and Analytical Support.*SOP# 16-DAT-01.00

Environmental Services Assistance Team (2012) *Surface Water Sampling*. SOP# FLD-1

Environmental Services Assistance Team (2012) *Shallow Stream Sediment Sampling.* SOP# FLD-6

Environmental Services Assistance Team (2012) *Global Positioning System (GPS) – Trimble GeoXT 2008 series.* SOP# FLD-7

Environmental Services Assistance Team (2012) *Water Quality Measurements with the In-Situ® Multi-Parameter Meter*. SOP# FLD-9

Environmental Services Assistance Team (2012) *Pore Water Sampling*. SOP# FLD-10

Environmental Services Assistance Team (2012) *Sample Custody and Labeling*. SOP# FLD-11

Environmental Services Assistance Team (2012) *General Field Sampling Protocols*. SOP# FLD-12

Environmental Services Assistance Team (2007) *Collection, Analysis, and Disposal of ESAT Laboratory Waste.* SOP# LAB 01.01

Environmental Services Assistance Team (2009) *Sample Receipt, Custody, Storage and LIMS Entry of Samples* SOP# LAB 05.02

TechLaw (2012) *Field Procedures - Analytical Support and Laboratory Selection.* SOP 02-06-05

Table

Table A.7-3 Water
QC Criteria

<table>
<tr><th>Target Analytes</th><th>EPA Method[1]</th><th>Instrument</th><th>Fraction Evaluated</th><th>Sample Volume Req'd (ml)</th><th>Preservation</th><th>Holding Time</th><th>Laboratory MDL, ug/L</th><th>Laboratory PQL, ug/L</th><th>CDPHE Surface Water Regulations[3]</th><th>MCL[4]</th></tr>
<tr><td>Aluminum (Al)</td><td>200.7</td><td>ICP-OE</td><td>TR & Diss</td><td rowspan="10">Diss - 250ml
TR - 500ml</td><td rowspan="20">HNO_3 to pH <2</td><td rowspan="11">180 days</td><td>20</td><td>50</td><td>750</td><td>-</td></tr>
<tr><td>Beryllium (Be)</td><td>200.7</td><td>ICP-OE</td><td>TR & Diss</td><td>2</td><td>5</td><td>-</td><td>4</td></tr>
<tr><td>Calcium (Ca)</td><td>200.7</td><td>ICP-OE</td><td>TR & Diss</td><td>100</td><td>250</td><td>NA</td><td>NA</td></tr>
<tr><td>Iron (Fe)</td><td>200.7</td><td>ICP-OE</td><td>TR & Diss</td><td>100</td><td>250</td><td>300 (WS)</td><td>300 (Diss only)</td></tr>
<tr><td>Chromium (Cr)</td><td>200.7</td><td>ICP-OE</td><td>TR & Diss</td><td>2</td><td>5</td><td>50 (TR)</td><td>100</td></tr>
<tr><td>Magnesium (Mg)</td><td>200.7</td><td>ICP-OE</td><td>TR & Diss</td><td>100</td><td>250</td><td>NA</td><td>NA</td></tr>
<tr><td>Manganese (Mn)</td><td>200.7</td><td>ICP-OE</td><td>TR & Diss</td><td>2</td><td>5</td><td>50 (WS)</td><td>50</td></tr>
<tr><td>Strontium (Sr)</td><td>200.7</td><td>ICP-OE</td><td>TR & Diss</td><td>2</td><td>10</td><td>NA</td><td>NA</td></tr>
<tr><td>Silica (SiO_2)</td><td>200.7</td><td>ICP-OE</td><td>TR & Diss</td><td>250</td><td>1000</td><td>NA</td><td>NA</td></tr>
<tr><td>Zinc (Zn)</td><td>200.7</td><td>ICP-OE</td><td>TR & Diss</td><td>10</td><td>20</td><td>65</td><td>5000</td></tr>
<tr><td>Calculated Hardness</td><td>2340B[2]</td><td>Calculated from 200.7, Ca & Mg</td><td>TR & Diss</td><td>-</td><td>-</td><td>-</td><td>(50)</td><td>-</td></tr>
<tr><td>Antimony (Sb)</td><td>200.8</td><td>ICP-MS</td><td>TR & Diss</td><td rowspan="9">Diss - 250ml
TR - 500ml</td><td rowspan="9">180 days</td><td>0.5</td><td>1</td><td>-</td><td>6</td></tr>
<tr><td>Arsenic (As)</td><td>200.8</td><td>ICP-MS</td><td>TR & Diss</td><td>0.5</td><td>2</td><td>50</td><td>50</td></tr>
<tr><td>Cadmium (Cd)</td><td>200.8</td><td>ICP-MS</td><td>TR & Diss</td><td>0.1</td><td>0.2</td><td>3.5</td><td>5</td></tr>
<tr><td>Copper (Cu)</td><td>200.8</td><td>ICP-MS</td><td>TR & Diss</td><td>0.5</td><td>1</td><td>7</td><td>1000</td></tr>
<tr><td>Lead (Pb)</td><td>200.8</td><td>ICP-MS</td><td>TR & Diss</td><td>0.1</td><td>0.2</td><td>1.2</td><td>50</td></tr>
<tr><td>Nickel (Ni)</td><td>200.8</td><td>ICP-MS</td><td>TR & Diss</td><td>0.5</td><td>1</td><td>29</td><td>100</td></tr>
<tr><td>Selenium (Se)</td><td>200.8</td><td>ICP-MS</td><td>TR & Diss</td><td>0.5</td><td>1</td><td>4.6</td><td>50</td></tr>
<tr><td>Silver (Ag)</td><td>200.8</td><td>ICP-MS</td><td>TR & Diss</td><td>0.2</td><td>0.5</td><td>0.1</td><td>100</td></tr>
<tr><td>Thallium (Tl)</td><td>200.8</td><td>ICP-MS</td><td>TR & Diss</td><td>0.1</td><td>0.2</td><td>15</td><td>500</td></tr>
<tr><td>Dissolved Organic Carbon</td><td>415.3</td><td>Combustion/Non-dispersive IR</td><td>Diss</td><td>250 ml</td><td>Phosphoric acid, pH<2</td><td>28 days</td><td>1 mg/L</td><td>1 mg/L</td><td>-</td><td>-</td></tr>
<tr><td>Alkalinity</td><td>310.1</td><td>Mettler AT</td><td>Total</td><td>250 ml</td><td>Cooled to 4°C +/- 2</td><td>14 days</td><td>5</td><td>5</td><td>NA</td><td>NA</td></tr>
<tr><td>Chloride</td><td>300.0[6]</td><td rowspan="3">Ion Chromatography</td><td>Diss</td><td rowspan="3">250 ml</td><td rowspan="3">Cooled to 4°C +/- 2</td><td rowspan="3">28 days</td><td>1.0 mg/L</td><td>2.0 mg/L</td><td>250 mg/L</td><td>250 mg/L</td></tr>
<tr><td>Fluoride</td><td>300.0[6]</td><td>Diss</td><td>0.1 mg/L</td><td>0.2 mg/L</td><td>2 mg/L</td><td>4 mgL</td></tr>
<tr><td>Sulfate</td><td>300.0[6]</td><td>Diss</td><td>2 mg/L</td><td>5 mg/L</td><td>250 mg/L</td><td>250 mg/L</td></tr>
</table>

Diss = Dissolved metals fraction, i.e. source water filtered through 0.45 um filter prior to preservation (acidified).

TR = Total recoverable metals, source water, acidified (preserved).

MDL: Method Detection Limit, statistically determined from the deviation in a series of seven low level (3-5x the anticipated MDL) analyses, treated exactly as unknown samples for analysis. 40 CFR Chapter 1, Part 136, Appendix B

PQL: Practical Quantitation Level. Target analyte concentrations between PQL and MDL qualified as estimated, 'J', due to potential high variability. 40 CFR Parts 9, 141 and 142 [WH-FRL-6934-9]

[1]EPA's *Methods for the Determination of Metals in Environmental Samples*, Supplement I, May 1994 (Series 200 Methods)

[2]*Standard Methods for the Examination of Water and Wastewater*, 18th Edition, 1992

[3] *Colorado Department of Public Health and Environment Water Quality Control Commission, Regulation 31, The Basic Standards and Methodologgies for Surface Water (5 CCR 1002-31)*, Effective March 22, 2005. Three different standards apply to the waters in th

[4] MCL: Maximum Contaminant Level, a concentration set by the above CDPHE publication, Table III, "Drinking Water Supply".

[5]EPA's *Test Methods for Determining Solid Waste*, SW-846

[6]EPA's *Methods for Chemical Analysis of Water and Wastes*, June 2003

[7]Water supply limits for organic chemicals taken from CDPHE EWQCC, Reg. 31. Lower value of given range is reported.

Table A.7-4 Sediment/Macroinvertebrates Contaminants of Concern, Detection Limits and Methods

Target Analytes	EPA Digestion Method[1]	EPA Method[1]	Instrument	Fraction Evaluated	Sample Volume	Preservation	Holding Time	Laboratory PQL (mg/kg)	Sediment Screening Benchmark (mg/kg)
Aluminum (Al)	200.2	200.7	ICP-OE	TR	200 grams	Ice	180 days	5	25,519
Beryllium (Be)	200.2	200.7	ICP-OE	TR				0.5	NA
Calcium (Ca)	200.2	200.7	ICP-OE	TR				25	NA
Chromium (Cr)	200.2	200.7	ICP-OE	TR				0.5	43
Copper (Cu)	200.2	200.7	ICP-OE	TR				1	32
Iron (Fe)	200.2	200.7	ICP-OE	TR				25	188,400
Magnesium (Mg)	200.2	200.7	ICP-OE	TR				25	NA
Manganese (Mn)	200.2	200.7	ICP-OE	TR				0.5	631
Zinc (Zn)	200.2	200.7	ICP-OE	TR				2	121
Antimony (Sb)	200.2	200.8	ICP-MS	TR				0.1	2
Arsenic (As)	200.2	200.8	ICP-MS	TR				0.2	9.8
Cadmium (Cd)	200.2	200.8	ICP-MS	TR				0.02	1
Lead (Pb)	200.2	200.8	ICP-MS	TR				0.02	36
Nickel (Ni)	200.2	200.8	ICP-MS	TR				0.1	23
Selenium (Se)	200.2	200.8	ICP-MS	TR				0.1	NA
Silver (Ag)	200.2	200.8	ICP-MS	TR				0.05	1
Thallium (Tl)	200.2	200.8	ICP-MS	TR				0.1	NA
Mercury (Hg)	7473	7473	CVAA	Total			28 days	0.02	0.18

Notes:

TR - Total recoverable metals from bulk sediment

PQL - Practical Quantitation Limit

mg/kg - milligrams per kilogram

Sample containers should be plastic and capable of holding the required sample volume.

[1]EPA's *Methods for the Determination of Metals in Environmental Samples*, Supplement I, May 1994 (Series 200 Methods)

[2]*Development and Evaluation of consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems.* Environ. Contam. Toxicol. Vol. 39, pp. 20-31. 2000.

[3]Not applicable - no established criteria for aluminum

[4]*Consensus-based sediment guidelines: Recommendations for use & application.* Wisconsin Dept. of Natural Resources, Madison, WI. Report No. WT-732-2003. December, 2003.

Table A.7-5
Upper Animas Mining District
Sampling Locations and Activities – April (deployment) and July (extraction) 2014

			Sediment	Surface Water		Pore Water
	Station Location	Site Description	Total Recoverable Metals and Mercury	MiniSipper	STIL	Dissolved Metals (Nitric)
Animas River	A55	Animas station at Howardsville Gauge	1	1		1
	A56	Animas above Arastra & Mill Overflow pipe (Lat 37.8278 Lon -107.6242)	1	1		1
	A58	Arastra @ confluence (Lat 37.8258 Lon -107.6242)	1			1
	A60	Animas below Arastra	1			1
	A61	Animas above Boulder	1			1
	A64	Animas below Boulder & Aspen tribs.	1			1
	A65	Animas opposite Power House	1			1
	A66	Animas @ Lakawanna bridge	1			1
	A67	Swansea @ confluence			10 Locations determined in field	
	A68	14th Street Gauge @ 13th Street Bridge	1	1		1
	A72	Animas Gauge below Silverton	1	1		1
	A73	Animas upstream of Elk Creek	1	1		1
	A73B	Animas downstream of Elk Creek	1			1
	A75D	Animas upstream of Cascade Creek	1	1		1
	A75B	Animas downstream of Cascade Creek	1			1
	A75CC	Cascade Creek @ confluence with Animas	1			1
	CB-Opp1	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1			
	CB-Opp2	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1			
	CB-Opp3	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1			
	CB-Opp4	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1			
	CB-Opp5	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1			
	Bbridge	Bakers Bridge	1	1		1
Mineral Creek	M34	Mineral Creek Gauge				1
	CC02H	CC above road to Mogul				
	CC01U	CC downstream of Sublevel 1 drainages				
	CC01T	CC downstream of Queen Anne				
	CC02B	Located on Cement Creek below the Mogul mine				
	CC03B	Cement Creek immediately upstream of Red and Bonita confluence. Site is straight across from a power pole. New site for June 2010. Site was named CCOPP-12 during sampling events previous to November 2010.				
	CC03	Cement Creek downstream of the Red and Bonita confluence and upstream of the North Fork confluence. Access site just upstream of the road crossing at the North Fork. New site for June 2010. Site was called CCOPP-11 in sampling events previous to November 2010.				
	CC18B	CC abv. Amer. Tunnel confluence, 2009				
	CC18	CC above treatment plant				
	CC21	CC below SF				
	CC21B	CC ups of Prospect Gulch and dws of Dry Gulch. Is CC21 different?				
	CC41	CC ups of Illinois Gulch and dws of Ohio Gulch				
	CC48	Cement Creek upstream from Animas				
	CC01C2	Grand Mogul consolidated discharges				
	CC02D	Mogul				
	CC02E	Gold Point				
	CC02K	Pride of Bonita				
	MTD-4	Mogul tailings drainage just upstream of confluence with Cement Creek. Site is upstream along Cement				
	FD-1	Fenn drainage upstream of confluence with Cement Creek. Site is near MTD-4 but downstream along Cement Creek.				
	CC03D	Red & Bonita @culvert				
	CC03C	Red & Bonita at outflow from mine tunnel.				
	CC19	American Tunnel				
	CC14	Silver Ledge				
	CC15	SF above Silver Ledge				
	CC16B	SF Below Silver Ledge				
	CC17	SF above CC				
	CC26	Prospect Gulch Mouth				
North Fork Cement Creek	CC04	NF CC above Gold King				
	CC07	NF Cement@rd crossing				
North Fork Cement Creek Tributaries	CC06	Gold King 7 level				
	CC06B	Second portal at the Gold King 7-Level mine. Site has considerably less flow than CC06 and is right beside a power pole. First sampled in Aug.2011				
		Total Number of Samples	21	7	10	17

Note: One duplicate sample will be collected for every 10 samples collected (10% frequency)

Field blank needed for each day of collection

* Indicates that sample is to be collected deemed conditions are safe and based on the judgement of the field sampling team

Table A.7-6
Upper Animas Mining District
Sampling Locations and Activities – May 2014

			Stream Measurements		Surface Water Collection			
	Station Location	Site Description	Temperature, Dissolved Oxygen, pH, and Conductivity	Stream Flows or gauge reading	Total Recoverable Metals (Nitric)	Dissolved Metals (Nitric)	DOC (Phosporic)	Anions and Alkalinity (Ice)
Animas River	A55	Animas station at Howardsville Gauge	1	1	1	1	1	1
	A56	Animas above Arastra & Mill Overflow pipe (Lat 37.8278 Lon -107.6242)	1	1	1	1	1	1
	A58	Arastra @ confluence(Lat 37.8258 Lon -107.6242)	1	1	1	1	1	1
	A60	Animas below Arastra	1	1	1	1	1	1
	A61	Animas above Boulder	1	1	1	1	1	1
	A64	Animas below Boulder & Aspen tribs.	1	1	1	1	1	1
	A65	Animas opposite Power House	1	1	1	1	1	1
	A66	Animas @ Lakawanna bridge	1	1	1	1	1	1
	A67	Swansea @ confluence	1	1	1	1	1	1
	A68	14th Street Gauge @ 13th Street Bridge	1	1	1	1	1	1
	A72	Animas Gauge below Silverton	1	1	1	1	1	1
	A73	Animas upstream of Elk Creek	1	1	1	1	1	1
	A73B	Animas downstream of Elk Creek	1	1	1	1	1	1
	A75D	Animas upstream of Cascade Creek	1	1	1	1	1	1
	A75B	Animas downstream of Cascade Creek	1	1	1	1	1	1
	A75CC	Cascade Creek @ confluence with Animas	1	1	1	1	1	1
	CB-Opp1	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1	1	1	1	1	1
	CB-Opp2	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1	1	1	1	1	1
	CB-Opp3	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1	1	1	1	1	1
	CB-Opp4	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1	1	1	1	1	1
	CB-Opp5	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1	1	1	1	1	1
	Bbridge	Bakers Bridge	1	1	1	1	1	1
Mineral Creek	M34	Mineral Creek Gauge	1	1	1	1		1
	CC02H	CC above road to Mogul	1	1	1	1		1
	CC01U	CC downstream of Sublevel 1drainages	1	1	1	1		1
	CC01T	CC downstream of Queen Anne	1	1	1	1		1
	CC02B	Located on Cement Creek below the Mogul mine	1	1	1	1		1
	CC03B	Cement Creek immediately upstream of Red and Bonita confluence. Site is straight across from a power pole. New site for June 2010. Site was named CCOPP-12 during sampling events previous to November 2010.	1	1	1	1		1
	CC03	Cement Creek downstream of the Red and Bonita confluence and upstream of the North Fork confluence. Access site just upstream of the road crossing at the North Fork. New site for June 2010. Site was called CCOPP-11 in sampling events previous to November 2010.	1	1	1	1		1
	CC18B	CC abv. Amer. Tunnel confluence,2009	1	1	1	1		1
	CC18	CC above treatment plant	1	1	1	1		1
	CC21	CC below SF	1	1	1	1		1
	CC21B	CC ups of Prospect Gulch and dws of Dry Gulch. Is CC21 different?	1	1	1	1		1
	CC41	CC ups of Illinois Gulch and dws of Ohio Gulch	1	1	1	1		1
	CC48	Cement Creek upstream from Animas	1	1	1	1		1
	CC01C2	Grand Mogul consolidated discharges	1	1	1	1		1
	CC02D	Mogul	1	1	1	1		1
	CC02E	Gold Point	1	1	1	1		1
	CC02K	Pride of Bonita	1	1	1	1		1
	MTD-4	Mogul tailings drainage just upstream of confluence with Cement Creek. Site is upstream along Cement	1	1	1	1		1
	FD-1	Fenn drainage upstream of confluence with Cement Creek. Site is near MTD-4 but downstream along Cement Creek.	1	1	1	1		1
	CC03D	Red & Bonita @culvert	1	1	1	1		1
	CC03C	Red & Bonita at outflow from mine tunnel.	1	1	1	1		1
	CC19	American Tunnel	1	1	1	1		1
	CC14	Silver Ledge	1	1	1	1		1
	CC15	SF above Silver Ledge	1	1	1	1		1
	CC16B	SF Below Silver Ledge	1	1	1	1		1
	CC17	SF above CC	1	1	1	1		1
	CC26	Prospect Gulch Mouth	1	1	1	1		1
North Fork Cement Creek	CC04	NF CC above Gold King	1	1	1	1		1
	CC07	NF Cement@rd crossing	1	1	1	1		1
North Fork Cement Creek Tributaries	CC06	Gold King 7 level	1	1	1	1		1
	CC06B	Second portal at the Gold King 7-Level mine. Site has considerably less flow than CC06 and is right beside a power pole. First sampled in Aug.2011	1	1	1	1		1
		Total Number of Samples	53	53	53	53	22	53

Note:One duplicate sample will be collected for every 10 samples collected (10% frequency)

Field blank needed for each day of collection

* Indicates that sample is to be collected deemed conditions are safe and based on the judgement of the field sampling team

Table A.7-7
Upper Animas
Sampling Locations and Activities – September 2014

			Stream Measurements		Surface Water Collection				Sediment	Pore Water	Macroinvertebrates	Fish[1]
	Station Location	Site Description	Temperature, Dissolved Oxygen, pH, and Conductivity	Stream Flows or gage reading	Total Recoverable Metals (Nitric)	Dissolved Metals (Nitric)	DOC (Phosphoric)	Anions and Alkalinity (Ice)	Total Recoverable Metals and Mercury	Dissolved Metals (Nitric)	Demographics	Population survey and tissues for TRM
Animas River	A55	Animas station at Howardsville Guage	1	1	1	1	1	1	1	1	1	1
	A56	Animas above Arastra & Mill Overflowpipe (Lat 37.8278 Lon -107.6242)	1	1	1	1	1	1	1	1	1	
	A58	Arastra @ confluence(Lat 37.8258 Lon -107.6242)	1	1	1	1	1	1	1	1	1	
	A60	Animas below Arastra	1	1	1	1	1	1	1	1		
	A61	Animas above Boulder	1	1	1	1	1	1	1	1		
	A64	Animas below Boulder & Aspen tribs.	1	1	1	1	1	1	1	1		
	A65	Animas opposite Power House	1	1	1	1	1	1	1	1		
	A66	Animas @ Lakawanna bridge	1	1	1	1	1	1	1	1	1	
	A67	Swansea @ confluence	1	1	1	1	1	1				
	A68	14th Street Gauge @ 13th Street Bridge	1	1	1	1	1	1	1	1	1	1
	A72	Animas Gauge below Silverton	1	1	1	1	1	1	1	1	1	
	A73	Animas Upstream of Elk Creek	1	1	1	1	1	1	1	1	1	1
	A73B	Animas downstream of Elk Creek	1	1	1	1	1	1	1	1	1	
	A75D	Animas upstream of Cascade Creek	1	1	1	1	1	1	1	1	1	1
	A75B	Animas downstream of Cascade Creek	1	1	1	1	1	1	1	1	1	
	A75CC	Cascade Creek @ confluence with Animas	1	1	1	1	1	1	1	1	1	
	CB-Opp1	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1	1	1	1	1	1	1			
	CB-Opp1	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1	1	1	1	1	1	1			
	CB-Opp1	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1	1	1	1	1	1	1			
	CB-Opp1	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1	1	1	1	1	1	1			
	CB-Opp1	Chicago Basin opportunistic sample location along Needle Creek to the confluence with Animas River	1	1	1	1	1	1	1			
	Bbridge	Bakers Bridge	1	1	1	1	1	1	1	1	1	
Mineral Creek	M34	Mineral Creek Gauge	1	1	1	1	1	1	1	1	1	t
	CC02H	CC above road to Mogul	1	1	1	1		1				
	CC01U	CC downstream of Sublevel 1drainages	1	1	1	1		1				
	CC01T	CC downstream of Queen Anne	1	1	1	1		1				
	CC02B	Located on Cement Creek below the Mogul mine	1	1	1	1		1				
	CC03B	Cement Creek immediately upstream of Red and Bonita confluence. Site is straight across from a power pole. New site for June 2010. Site was named CCOPP-12 during sampling events previous to November 2010.	1	1	1	1		1				
	CC03	Cement Creek downstream of the Red and Bonita confluence and upstream of the North Fork confluence. Access site just upstream of the road crossing at the North Fork. New site for June 2010. Site was called CCOPP-11 in sampling events previous to November 2010.	1	1	1	1		1				
	CC18B	CC abv. Amer. Tunnel confluence,2009	1	1	1	1		1				
	CC18	CC above treatment plant	1	1	1	1		1				
	CC21	CC below SF	1	1	1	1		1				
	CC21B	CC ups of Prospect Gulch and dws of Dry Gulch.	1	1	1	1		1				
	CC41	CC ups of Illinois Gulch and dws of Ohio Gulch	1	1	1	1		1				
	CC48	Cement Creek upstream from Animas	1	1	1	1	1	1	1			
	CC01C2	Grand Mogul consolidated discharges	1	1	1	1		1				
	CC02D	Mogul	1	1	1	1		1				
	CC02E	Gold Point	1	1	1	1		1				
	CC02K	Pride of Bonita	1	1	1	1		1				
	MTD-4	Mogul tailings drainage just upstream of confluence with Cement Creek. Site is upstream along Cemen	1	1	1	1		1				
	FD-1	Fenn drainage upstream of confluence with Cement Creek. Site is near MTD-4 but downstream along Cement Creek.	1	1	1	1		1				
	CC03D	Red & Bonita @culvert	1	1	1	1		1				
	CC03C	Red & Bonita at outflow from mine tunnel.	1	1	1	1		1				
	CC19	American Tunnel	1	1	1	1		1				
	CC14	Silver Ledge	1	1	1	1		1				
	CC15	SF above Silver Ledge	1	1	1	1		1				
	CC16B	SF Below Silver Ledge	1	1	1	1		1				
	CC17	SF above CC	1	1	1	1		1				
	CC26	Prospect Gulch Mouth	1	1	1	1		1				
North Fork Cement Creek	CC04	NF CC above Gold King	1	1	1	1		1				
	CC07	NF Cement@rd crossing	1	1	1	1		1				
North Fork Cement Creek Tributaries	CC06	Gold King 7 level	1	1	1	1		1				
	CC06B	Second portal at the Gold King 7-Level mine. Site has considerably less flow than CC06 and is right beside a power pole. First sampled in Aug.2011	1	1	1	1		1				
		Total Number of Samples	53	53	53	53	24	53	23	17	13	4

Note: One duplicate sample will be collected for every 10 samples collected (10% frequency)

Field blank needed for each day of collection

* Indicates that sample is to be collected deemed conditions are safe and based on the judgement of the field sampling team

[1] Sample locations are approximate

Table A.7-8
Upper Animas 2014
Sampling Location Coordinates

	Station Location	Latitude	Longitude
Animas River	A55	37.8286	-107.6083
	A56	37.8278	-107.6242
	A58	37.8258	-107.6242
	A60	37.8269	-107.6277
	A61	37.8253	-107.6305
	A64	37.8231	-107.6352
	A65	37.8208	-107.6416
	A66	37.8167	-107.6472
	A67	37.8144	-107.6527
	A68	37.8111	-107.6586
	A72	37.79027049	-107.6675778
	A73	37.72215833	-107.65482777
	A73B	37.72156666	-107.65443055
	A75D	37.59793423	-107.77532681
	A75B	37.59731135	-107.77683908
	A75CC	37.59824909	-107.77610081
	Bbridge	37.45890000	-107.79955000
Mineral Creek	M34	37.8028	-107.6722
Cement Creek	CC02H	37.91068611	-107.63409167
	CC01U	37.91074166	-107.6348528
	CC01T	37.91022500	-107.6335833
	CC02B	37.90873753	-107.6421324
	CC03B	37.89777777	-107.645925
	CC03	37.89553888	-107.6470056
	CC18B	37.89422919	-107.6472546
	CC18	37.89127189	-107.6491833
	CC21	37.88946461	-107.6542211
	CC21B	37.88252281	-107.667432
	CC41	37.8517	-107.6758
	CC48	37.81779722	-107.661803
Cement Creek Tributaries	CC01C2	37.91011944	-107.6329167
	CC02D	37.9098	-107.6384
	CC02E	37.90822992	-107.6384231
	CC02K	37.90749722	-107.640225
	MTD-4	37.90876111	-107.6417528
	FD-1	37.90868888	-107.6420972
	CC03D	37.8968	-107.6449
	CC03C	37.8972	-107.6439
	CC07	37.8951	-107.6468
	CC19	37.89098119	-107.6484397
	CC14	37.5236	-107.3838
	CC15	37.8756	-107.6439
	CC16B	37.87694341	-107.6451878
	CC17	37.8894	-107.6506
	CC26	37.88261596	-107.66774
North Fork Cement Creek	CC04	37.8944	-107.6328
	CC07	37.8951	-107.6468
North Fork Cement Creek Tributaries	CC06	37.8946	-107.6384
	CC06B	37.89473056	-107.6386222

Table B.1-1 Sampling Checklist

1. Make sure the necessary paperwork is in place for a field event: Approved LSR, SAP, and QAPP.

2. Coordinate sampling dates and times with members of the field team and talk with chemists involved in the project to see if your plans work for them. Coordinate sample delivery with outside laboratories.

3. Fill out the necessary paperwork: Comp Time forms and TAs if travel will be more than 50 miles from the laboratory. Be sure to have reservations made for airlines and hotels if necessary.

4. Make necessary arrangements with people outside of the Region VIII laboratory that are involved with the project. Arrange meeting times and places, vehicle needs, sampling teams, additional equipment needs, etc.

5. Inform any volunteers outside of the EPA laboratory group what will be involved with sampling - physical stressors, equipment to bring, lunch, water, etc.

6. Calibrate meters needed for fieldwork well-before leaving. Make sure:
   a. pH probes are filled.
   b. DO membranes are intact.
   c. Spare batteries, calibration logs, and pens are available for each meter.
   d. Replace pH and conductivity calibration standards with fresh solution.
   e. Condition new probes and replace damaged ones as needed. Buy new equipment from a scientific vendor if necessary.

7. Lay out needed sampling equipment in the field room (see attached list).

8. Check vehicles: fill with gas, top off windshield wiper fluid, equip with cell phones, walkie-talkies, and chargers.

9. Charge batteries for needed sampling equipment one or two nights before leaving: digital camera, hydrolab, GPS units, walkie-talkies, etc.

10. Pack vehicles the night before leaving. In the event of hot or cold weather, leave meters and deionized water in the field room and pack the day you leave.

11. In the event of a day-trip, calibrate meters the morning you leave.

Table B.1-2 Field Equipment List

Sample Containers:
250 ml HDPE containers
Gallon cubitainers
VOA vials
Glass Amber (BNA, pest)
1 oz plastic (sed. metals)

Filter Apparatus:
250 or 500 ml filters
Filter Stands
Vacuum pump with spare
Prefilters
Teflon Tweezers

Preservatives:
HNO_3 - metals
H_2SO_4 - nutrients
Phosphoric - TOC/DOC
HCl - VOAs (pipet)
$CaCO_3$ Acid Waste
    Container
Ice/Snow

Paperwork:
Flow forms with clipboard
SAP / HSP
Maps/Gazeteer
Chains
Tags
Field Notebook(s)
FedX Forms
Pens
Markers
Custody Seals

Meters (w/logs):
Flow meter-
    Tape measure Wading rod
    D batteries
    Rebar
    Forms
    Calculator Stopwatch, bucket
    Flumes- bubble level, shovel
GPS Units (charged)-
    Compass
    Distance meter
Digital Camera w/batteries
Multimeters (charged)-
    Cal standards
    Cap & cal. cup
    Membranes
    Fill solutions
    Control unit
    Sonde
    Logbook
    Cables - long/short

Misc:
Battery charger
pH test strips
Latex gloves
Neoprene gloves
Safety glasses
Kimwipes
Trash bags
Plastic Bags
Tape
Bucket
Coolers
DI rinse bottles
Cell Phones w/ charger
Walkie-Talkies w/batteries
Shovel
Spare car keys
Vehicle log & credit card
Govt. purchase card
DI water for blanks
DI rinse bottles

Macroinvertebrate Samples:
Sample containers
D-Frame Dip Net
Surber Sampler
Sieve
White Tray
Rinse bottle
Picking forceps/brush
95% Ethanol preservative
Bucket
Orange water gloves

Well Sampling:
Solinst depth meter w/battery
Grundfos well pump-
    Control unit
    Hose reel w/ pump
    Discharge tube
    Cooling sleeve
    Screw driver
    Wrenches
    Metric hex keys
    DI water
    3 to 2 prong electrical converter
Generator- gas, ext. chord
Well Bailors
String or chord
Long multimeter cable

Summer Field Gear:
Backpacks
Hiking Boots
Hat
GorTex Waders
Wading Boots
Rain Parka
Wool Socks
Layered Clothing
Sunscreen
Chapstick
Bug Spray
Sun Glasses
Water/Food
Pocket Knife

Winter Field Gear:
Shovel/Ice Breaker
Backpacks
Snowshoes
Hiking Poles
Insulated Water Gloves
Hat
Gloves
Balaclava
Neoprene Waders
Wading Boots
Wool Socks
Layered Clothing
Sunscreen
Chapstick
Sunglasses
Water/Food
Pocket Knife

Field Meters (when not using multimeters):
logbooks
pH-   buffers
    probe solutions batteries
DO-   Spare membranes filling solution
    Barometer
    Calibration equip:
    Winkler Bottle
    Starch
    0.035N Na Thio
    Buret/Pipet
    Buret Holder
    Flask w/ stir bar
    Powder Pillows:
    MnSO4
    Alk Iodide-Azide
    Sulfamic Acid
Conductivity-
    calibration stds

Table B.2-1
Filling Time for the DH-81 Sampler using a 1 liter Bottle

Velocity, ft/sec	Volume, mL	3/16-in nozzle	1/4-in nozzle	5/16-in nozzle
1.4	800		59	
1.6	800		52	
1.8	800		46	
2.0	800	74	41	27
2.2	800	67	38	24
2.4	800	61	35	22
2.6	800	57	32	20
2.8	800	53	30	19
3.0	800	49	28	18
3.2	800	46	26	17
3.4	800	43	24	16
3.6	800	41	23	15
3.8	800	39	22	14
4.0	800	37	21	13
4.2	800	35	20	13
4.4	800	33	19	12
4.6	800	32	18	12
4.8	800	31	17	11
5.0	800	29	17	11
5.2	800	28	16	10
5.4	800	27	15	10
5.6	800	26	15	9
5.8	800	25	14	9
6.0	800	25	14	9
6.2	800	24	13	9
6.4	800		13	8
6.6	800		13	8
6.8	800		12	8
7.0	800		12	8
7.2	800		12	
7.4	800		11	
7.6	800		11	

Table B.5-1
ESAT Region 8 - Metals QC Criteria

QC Check / Symbol	Explanation	Run Frequency	AcceptanceCriteria	CorrectiveAction
Initial Calibration Verification (ICV)	Certified standard or standard from a different lot/source than calibration standards	Beginning of run to verify calibration	90-110% recovery (%R) of "true value"	Terminate analysis, restandardize
Continuing Calibration Verification (CCV)	Approximate mid-range standard made from working standards stock	Every 10 unknowns and at end of run	90-110%R "True" value	Re-analyze immediately (once). Then: Restandardize and rerun all samples following last "acceptable" CCV. If recovery >110% and <120% and all associated samples (same analyte) show non-detected, no action required.
Spectral/Mass Interference Check for ICP-OE & ICP-MS (ICSA / ICSAB)	Analyze spectral interferents at high concentrations alone (ICSA) and with other target analytes (ICSAB) to evaluate the effect on analyte recovery	Once per analytical run, prior to sample analyses	ICSAB: ± 20%R 'true value' ICSA: ± 20%R 'true value' or < ±PQL whichever is greater	Evaluate the sample analyte levels. Rerun ICSA/AB or use an alternate wavelength. If interferent levels in the samples don't approach ICSA interferent levels, no action is required. If necessary, recalculate IECs & rerun associated samples
Calibration Blanks, Initial & Continuing (ICB & CCB)	Blank with same reagents as working standards; i.e. zero point on curve	Beginning, end, and after each ICV/CCV during analytical run	≤ ±PQL	Re-analyze immediatelyonce. If still unacceptable, terminate analysis & restandardize. Rerun all samples analyzed after last "acceptable" blank. Evaluate interferent level(s) vs samples, use prof judgement for addit'l required sample reruns.
Preparation Blank (PB)	Digested or prepared blank processed identical to samples. Aliquot of clean water prepared using same reagents/volumes as unknown samples.	Once per preparation batch/per matrix, or at 5% frequency, whichever is greatest	≤ ±PQL	PB > PQL: Redigest all samples >MDL and <10x PB value PB < -PQL: Re-calibrate and re-analyze all associated samples
Matrix Spike & Matrix Spike Duplicate (MS & MSD)	Unknown sample (NOT a field blank) fortified at approximately 10-100x MDL for each target analyte. High concentration samples (spike <25% sample target analyte concentration), no calculation is required	1 per 20 unknowns per matrix, whichever is greatest (One PB Spike per PB)	Spike recovered at: 80-120% (ICP& MS) - waters 65-135% (all) - solids	Compose 1 post-digest spike (PS) and retest, note in the narrative. (Analyze original sample with PS) Evaluate duplicate reproducibility. Compare results to LFB/PBS for similar trends. If no similar trends observed, assume a matrix effect. Qualify correspondinganalyte data as estimated 'J' for similar matrix samples in set.
Lab Fortified Blank (LFB or PBS)	Spike of reagent blank at same level as MS (analyze/prep identical to samples)	Recommend: once/run	85-115%R of expected (for targe analytes)	Used for comparison to Matrix Spike. If MS/MSD in-control no corrective action necessary.
Lab Control Sample (LCS)	For solid & liquid digested samples. A known of similar matrix prepared the same as unknown samples.	1 per prep batch or one per matrix, whicheveris greater.	Aq: 80-120%R of "true" Solids: 70-130%R of "true" or publishedlimits	Recalibrate & reanalyze. If still unacceptable, check for corresponding high or low results in pre-digest spikes, if similar, redigest all associated samples
Serial Dilution (L)	Sample analyzed at 5x the reported analysis. (for matrix effect evaluation) Applies to analytes >50x MDL (in the original analyzed solution)	1 per 20 unknown	Diluted value 90-110% of original analysis.	Concentrations compared/reported from the analyzed solution only. Check IECs and re-analyze. May re-analyze both sample and 'L' at a higher dilution. Use professional judgement, and discuss outliers in the narrative.
Detection Limit Standard (CRI/CRA)	Low level standard ≈3-5x MDL concentration. Applies to all target analytes except Al, Ca, Fe, Mg, Na, & K	Once per analytical batch prior to unknowns	50-150%R for Sb, Pb, and Tl. 70-130%R for other target analytes*.	1. Rerun 2. If all associated samples ≥CCV for outlier analyte, no action required 3. Correct instrument's sens. problem or else need to redetermine and raise reporting limits *[Al, Ca, Fe, Mg, Na, & K are monitored without corrective actions]
ICP-MS Internal Standard (IS)	IS standard solution added to all samples, blanks, and standards.	All samples and standards corrected for IS response.	60% - 125%R of IS associated with target analyte(s)	[IS recovery determined versus calibration blank response.] Dilute sample by 2, re-analyze. Continue to dilute until IS %R acceptable.

Table B.5-2
QA/QC Calculation Algorithms

Statistical QC Parameter Evaluated	Acronym	Analyses Applied to	Calculation Algorithm
Percent Recovery	%R	Spike recovery determinations	$\%R = ((C_s - S_a) \div (S_a)) \times 100$
Percent Recovery	%R	ICV/CCV, ICSAB, LCS	$\%R = (A_T \div T) \times 100$
Relative Percent Difference	RPD	Variance between duplicates	$RPD = ((C - C_D) / ((C + C_D) \div 2)) \times 100$
Percent Difference	%D	Serial dilution variance	$\%D = ((C - C_L) / C) \times 100$

Notes:
C = Sample extract concentration
C_s = Sample extract, spiked concentration
S_a = Spike amount added
T = True (possibly certified) amount in standard solution
Hardness = (Ca, mg/L)*2.497 + (Mg, mg/L)*4.118
C_D = Duplicate sample concentration
C_L = Sample extract concentration, dilution factor corrected.
AT = Analyzed concentration for the known standard.

Figures

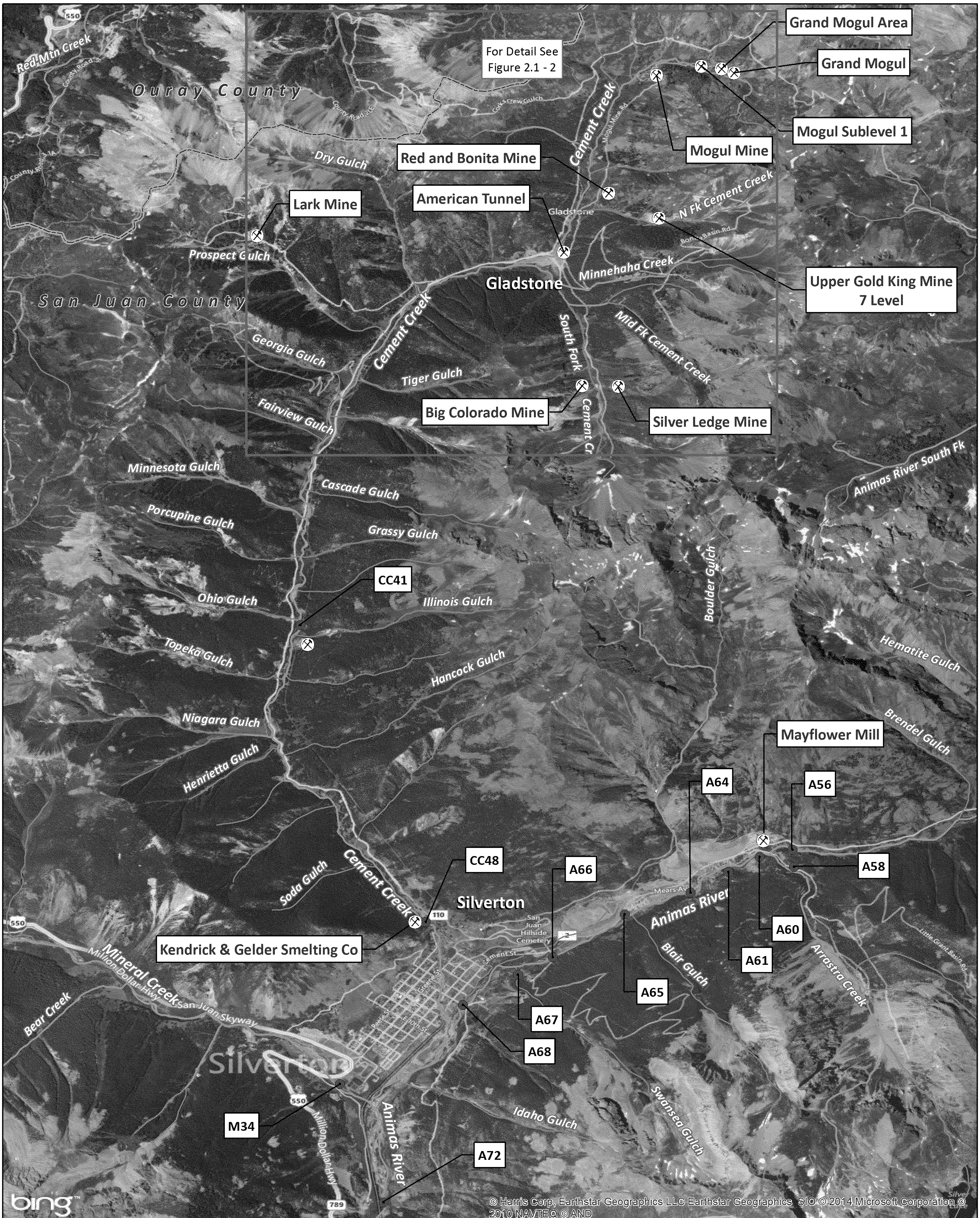


Figure A.7-1
Upper Animas Mining District Area Overview
Upper Animas River and Cement Creek, Silverton, CO

TechLaw

Sample Locations

Mine Locations

Rivers and Streams

County Boundaries

Date: March 18, 2014

Data Sources:
Sample Locations: U.S. EPA Region 8 (2013)
Mine Locations: U.S. EPA and ESAT (2012)
Rivers and Streams: CDOW 1:24k (2004)
County Boundaries: U.S. Census Bureau (2011)
Image: Microsoft Bing web service (2014)

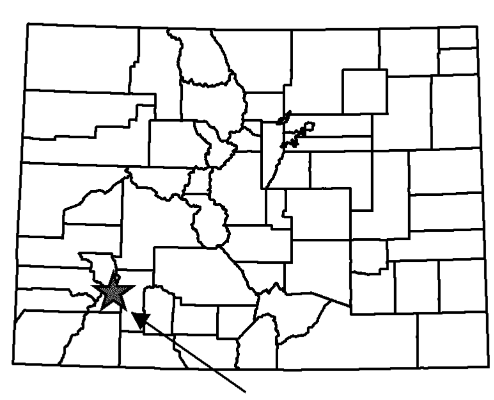
Coordinate System/Projection:
UTM Zone 13 North, NAD 83, Meters

N
W E
S

0 0.5 1 Miles

0 0.5 1 Kilometers

Colorado



Area of Interest

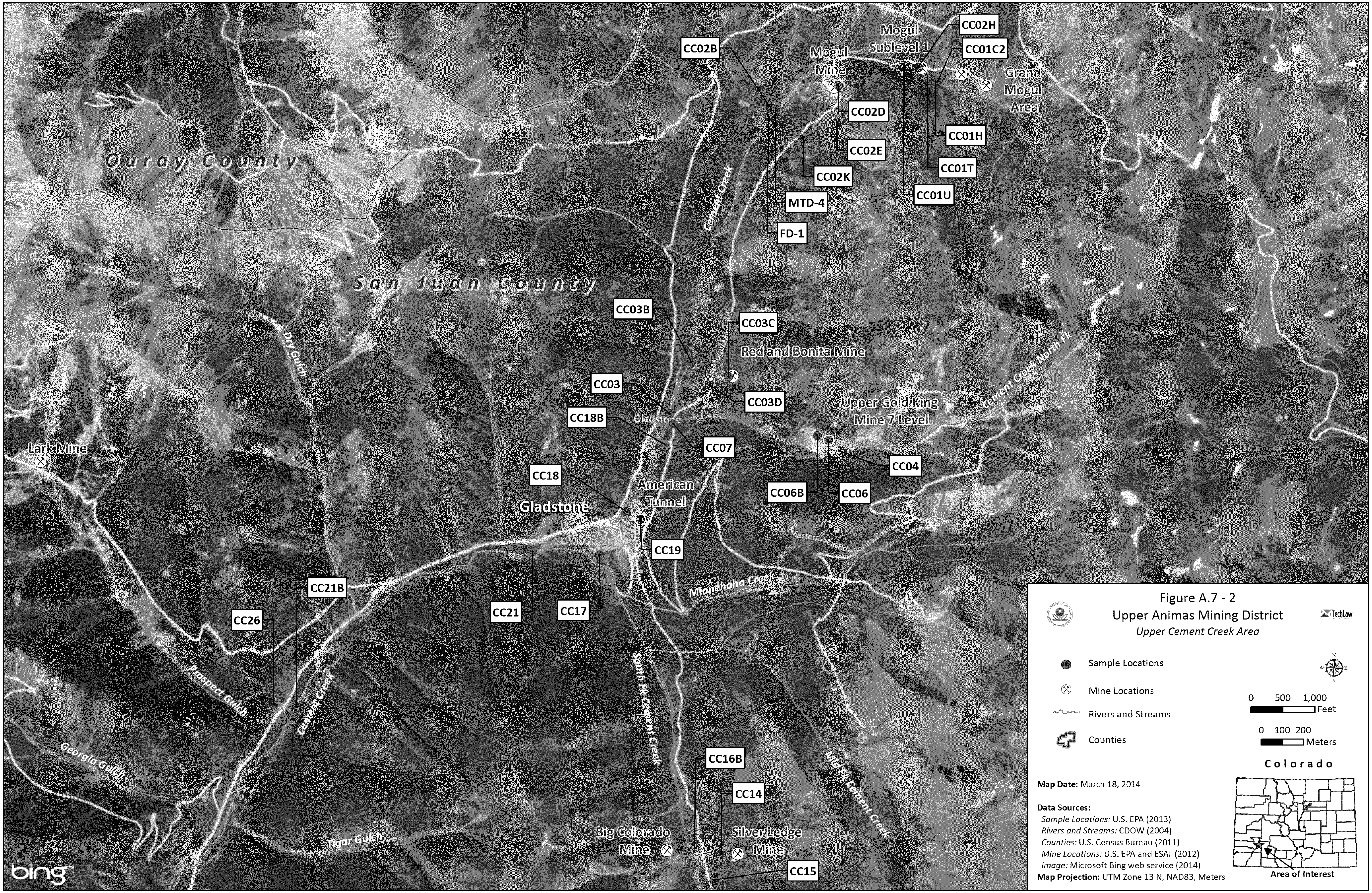


Figure A.7 - 2
Upper Animas Mining District
Upper Cement Creek Area
TechLaw
Sample Locations
Mine Locations
Rivers and Streams
Counties
0 500 1,000 Feet
0 100 200 Meters
Colorado
Area of Interest
Map Date: March 18, 2014
Data Sources:
Sample Locations: U.S. EPA (2013)
Rivers and Streams: CDOW (2004)
Counties: U.S. Census Bureau (2011)
Mine Locations: U.S. EPA and ESAT (2012)
Image: Microsoft Bing web service (2014)
Map Projection: UTM Zone 13 N, NAD83, Meters
Ouray County
San Juan County
Mogul Mine
Mogul Sublevel 1
Grand Mogul Area
CC02H
CC01C2
CC02B
CC02D
CC02E
CC01H
CC02K
CC01T
MTD-4
CC01U
FD-1
Corkscrew Gulch
County Road
Cement Creek
CC03B
CC03C
Mogul Mine Rd
Red and Bonita Mine
CC03
CC03D
Cement Creek North Fk
Bonita Basin
Upper Gold King Mine 7 Level
CC18B
Gladstone
CC07
CC04
Lark Mine
Dry Gulch
CC18
American Tunnel
CC06B
CC06
Gladstone
CC19
Eastern Star Rd
Bonita Basin Rd
Minnehaha Creek
CC21B
CC26
CC21
CC17
Prospect Gulch
South Fk Cement Creek
Georgia Gulch
CC16B
CC14
Mid Fk Cement Creek
Big Colorado Mine
Silver Ledge Mine
Tigar Gulch
CC15
bing

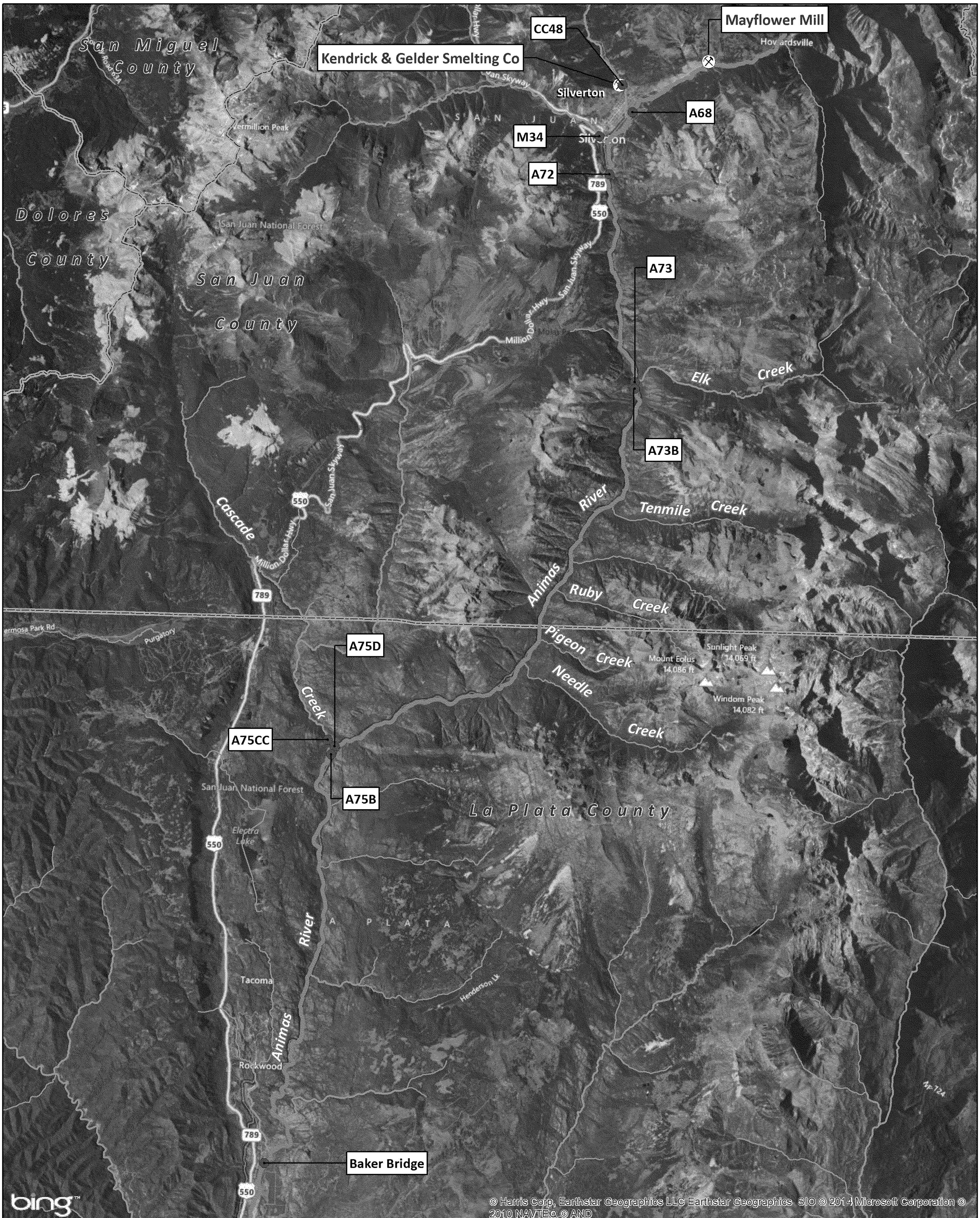


Figure A.7-3
Upper Animas Mining District
Animas River Canyon Area

- Sample Locations
- Mine Locations
- Rivers and Streams
- County Boundaries

Date: March 18, 2014

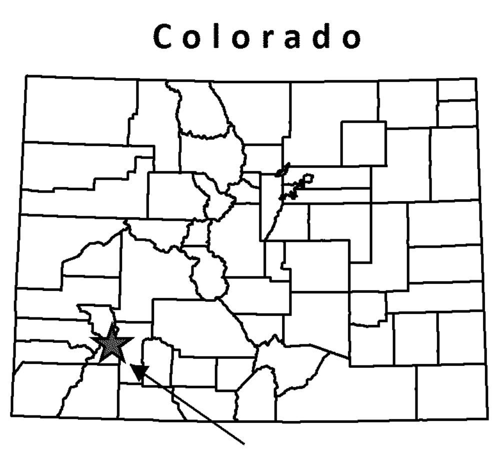
Data Sources:
Sample Locations: U.S. EPA Region 8 (2013)
Mine Locations: U.S. EPA and ESAT (2012)
Rivers and Streams: CDOW 1:24k (2004)
County Boundaries: U.S. Census Bureau (2011)
Image: Microsoft Bing web service (2014)

Coordinate System/Projection:
UTM Zone 13 North, NAD 83, Meters

TechLaw

0 1 2 Miles

0 1 2 Kilometers



Appendix A
Standard Operating Procedures

16-DAT-01.00
Data Management for Field Operations and Analytical Support

Technical Approval: Mark McDaniel

PRINT NAME

SIGNATURE **Date:** 3/12/14

QA Management Approval: Nikki MacDonald

PRINT NAME

SIGNATURE **Date:** 03/12/14

This document was prepared by the ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of ESAT support services. This is a controlled document and may only be provided to a third party, such as consultants or other government agencies, at the direction of EPA and if all said third party recipients agree that the contents of this document remain confidential. If the document is provided as a controlled document, the user agrees to surrender the document upon request of EPA or ESAT Region 8. If the document is provided as an uncontrolled document, the user understands that subsequent revisions may not be provided.

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1.0 SOP Description

The purpose of this standard operating procedure (SOP) is to provide a consistent format for all Region 8 Environmental Services Assistance Team (ESAT) data management personnel who perform uploads to Scribe and management of associated databases and reports.

This SOP is applicable to all ESAT personnel who prepare, process, review, and load analytical data into the Scribe database for the Field Operations Group and the Analytical Support and Data Validation Group.

2.0 Abbreviations and Acronyms

EDD	Electronic Data Deliverable
ERT	Environmental Response Team
ESAT	Environmental Services and Assistance Team
LIMS	Laboratory Information Management System
SOP	Standard Operating Procedure
TDF	Technical Direction Form
TO	Task Order
USEPA	United States Environmental Protection Agency

3.0 Health and Safety

All office-related safety precautions must be followed. Consideration is given to ergonomics for staff members using a keyboard and sitting in front of a computer terminal for extended periods of time and all other work conditions where ergonomics may be an issue.

4.0 Equipment and Supplies

Standard office supplies are required for this SOP, such as a personal computer and central filing system. Specific equipment and supplies are listed below:

- Internet connection and access to the ESAT network drive
- Access to Scribe and the Scribe databases associated with Technical Direction Forms (TDFs) issued by the client
- Login capabilities to the Scribe.NET website
- Microsoft Office software applications
- External hard drive containing the appropriate databases for upload

5.0 General Procedures

ESAT personnel are responsible for acquiring, compiling, reviewing, and loading analytical data into the appropriate Scribe database associated with a specific TDF. Electronic Data Deliverables (EDDs) are posted to the network drive from the Laboratory Information Management System (LIMS) on or before the EDD due date.

5.1 Review, Obtain, and Prepare EDDs to be uploaded to Scribe

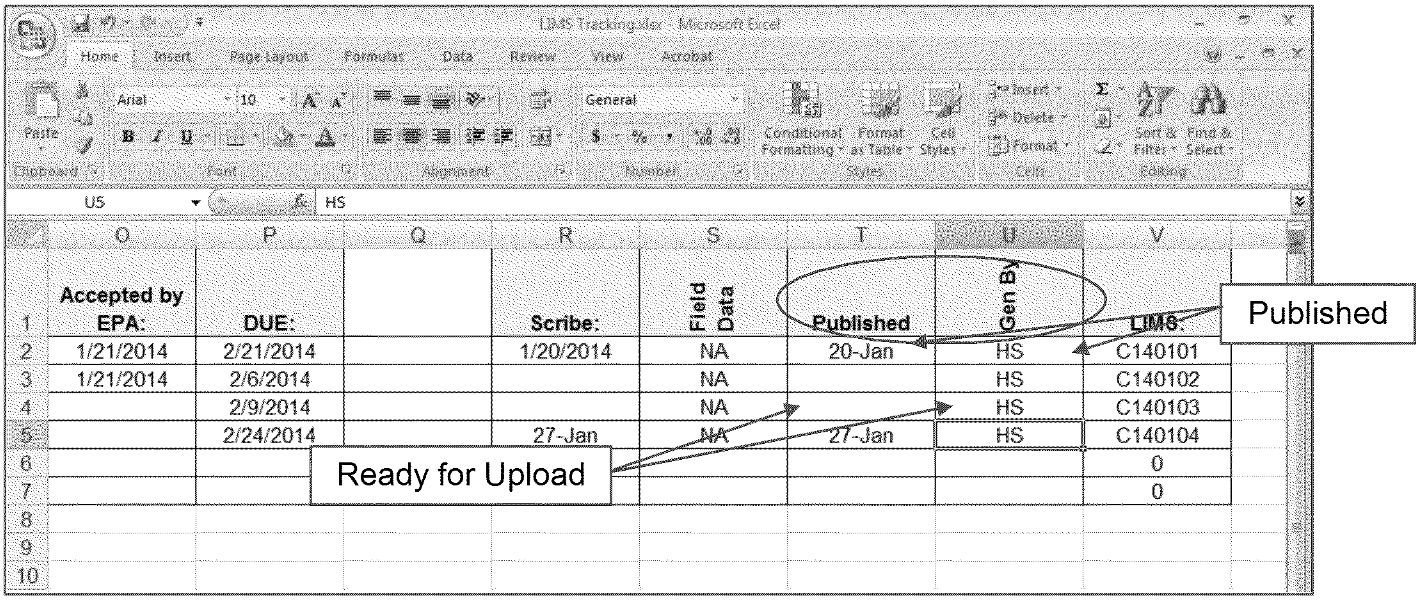
The analysts and/or Data Package Coordinator posts EDDs to the appropriate Task Order (TO) and project folder on the network drive upon completion. Prior to uploading or publishing a project to Scribe, review the LIMS Tracking spreadsheet to ensure that the

Data Package Coordinator has completed assembly and finalization of the current Sample Event(s) (Figure 5.2). EDDs that are complete and ready for upload are listed in the LIMS Tracking spreadsheet (located on the network drive), and will be signed off in the “Gen By” (generated by) column (Figure 5.1). EDDs that have been published will contain a date in the “Published” column (Figure 5.1).

Note: TO Numbers and TDFs are not permanent, and are subject to change based on the contract year, as well as the type and number of sample events.

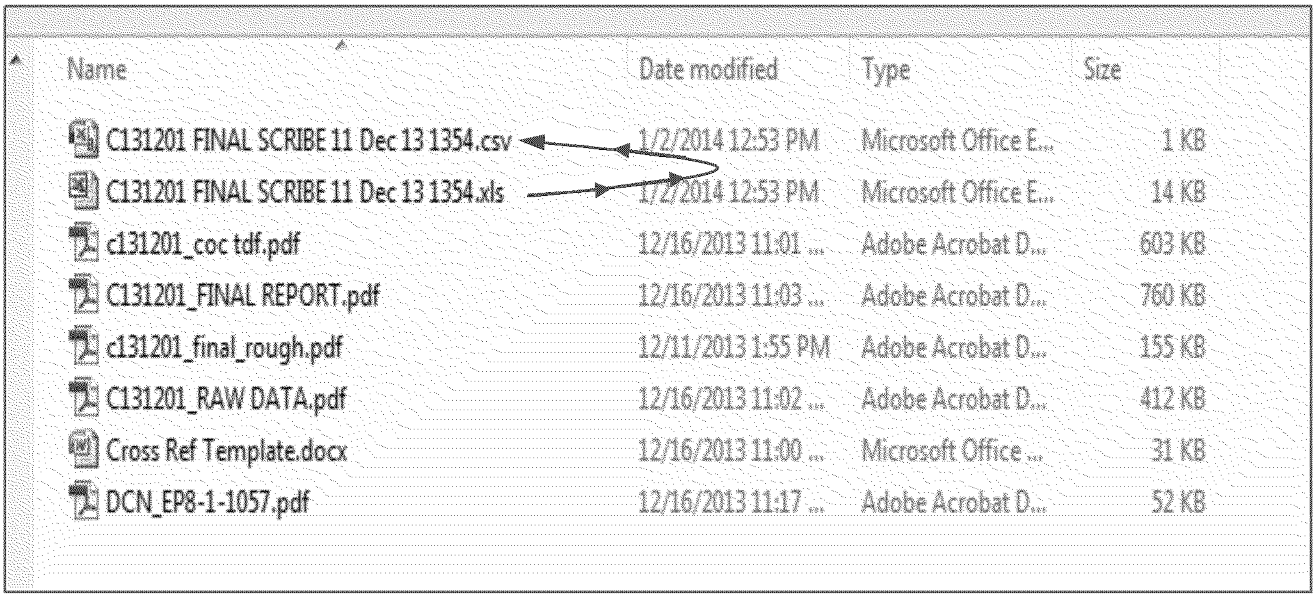
- Review the "Gen By" and "Published" columns. If the Data Package Coordinator's initials are listed in the "Gen by" column, but a date is not included in the "Published" column, that sampling event is ready for uploading to the database.

Figure 5.1 LIMS Tracking Spreadsheet Example



- Navigate to the TO and Projects to be uploaded
- Select the .xlsx EDD files, and within the same folder, convert them to .csv format for uploading to Scribe
- Repeat with all other EDDs noted from the LIMS Tracking spreadsheet

Figure 5.2 Example of Sample Event Folder from the Network Drive

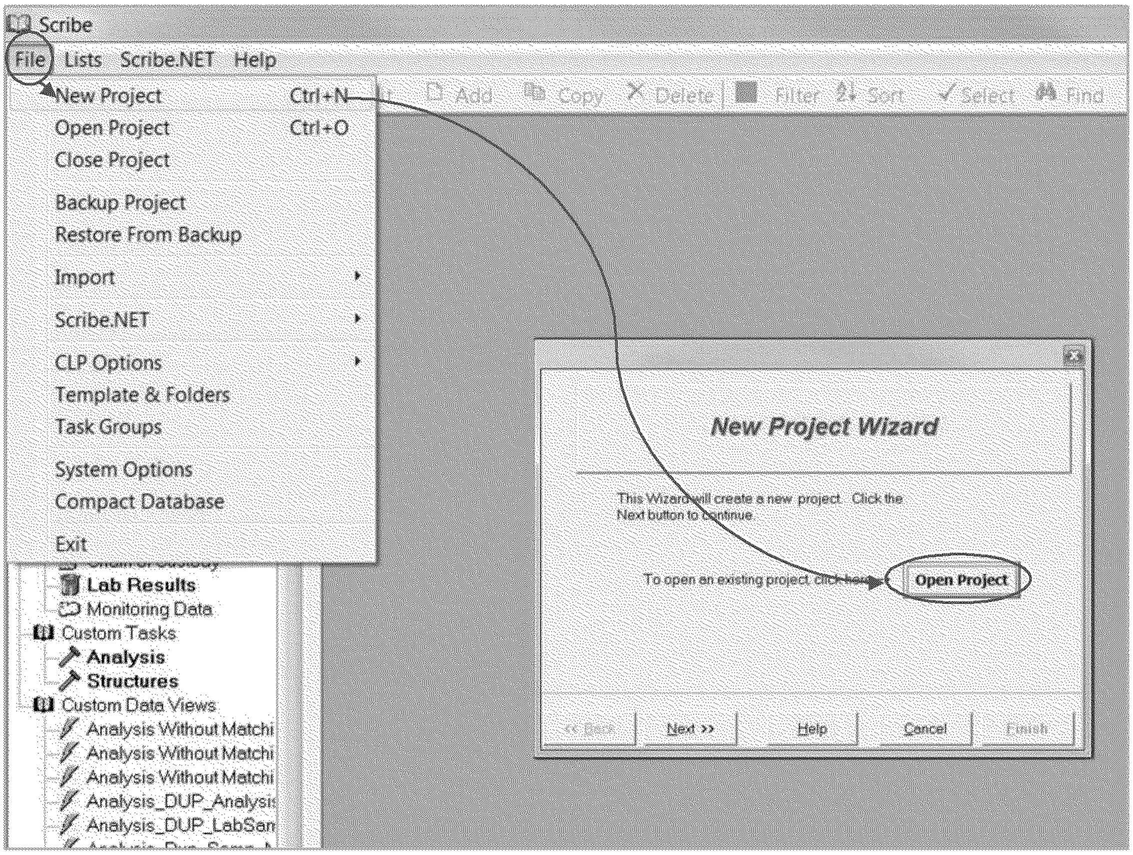


5.2 Scribe Data Load

Each TO project/TDF has its own database. Several separate sample events may occur under each project and will be managed according to the associated TO and project.

- Access the external hard drive containing the appropriate TO databases
- Open the Scribe Database Program
- Select "File" from the menu then "New Project"
- Select "Open Project" in the "New Project Wizard" dialogue box (this will open a Windows Explorer window from which to choose the appropriate folder pathway and database file)

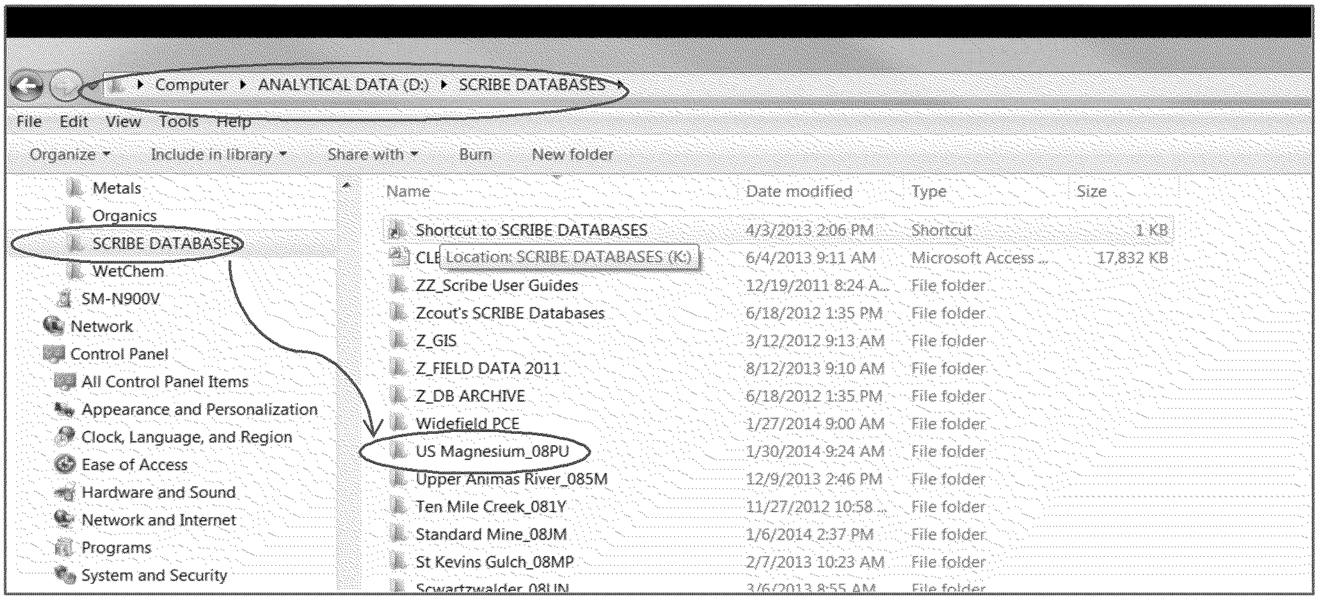
Figure 5.3 Scribe Database "Open Project"



- Navigate to the current TO and Project to be uploaded

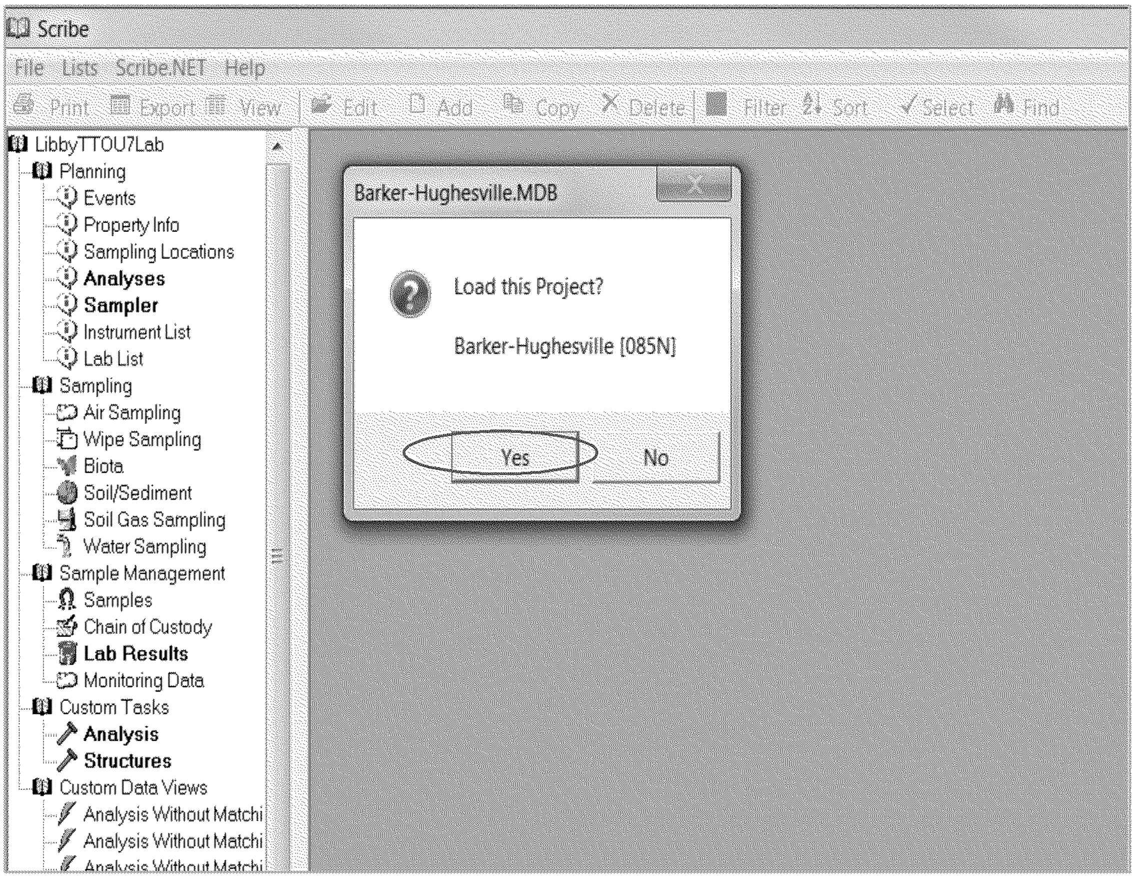
  Note: Databases for the current TO are stored on an external hard drive as shown in Figure 5.4, but the external storage location, pathways, and TO numbers are subject to change.

Figure 5.4 Scribe Database Project Pathway



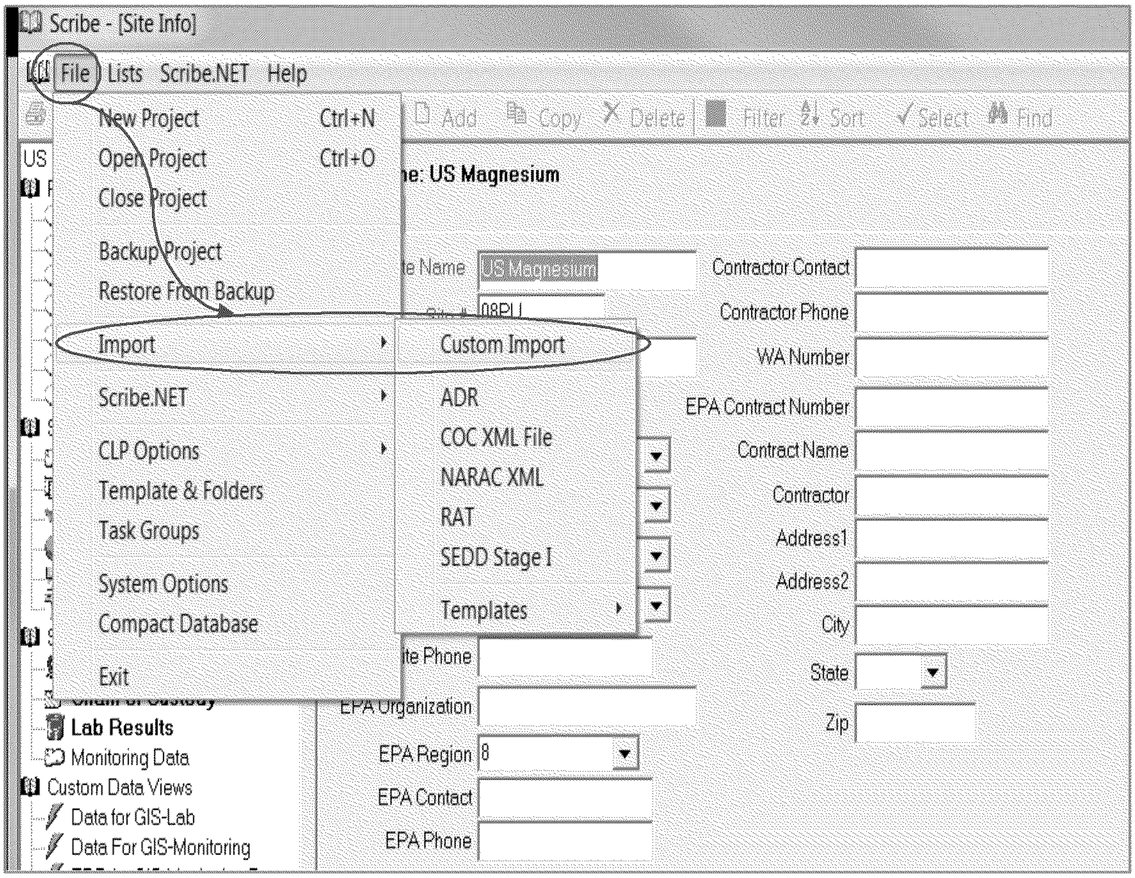
- Select "Yes" in the "Load this Project" dialogue box

Figure 5.5 Scribe Database Load Project



- Select "File -> Import -> Custom Import"
- Select "No" in the resulting "Backup Now?" dialogue box

Figure 5.6 Scribe "File/Import/Custom Import"

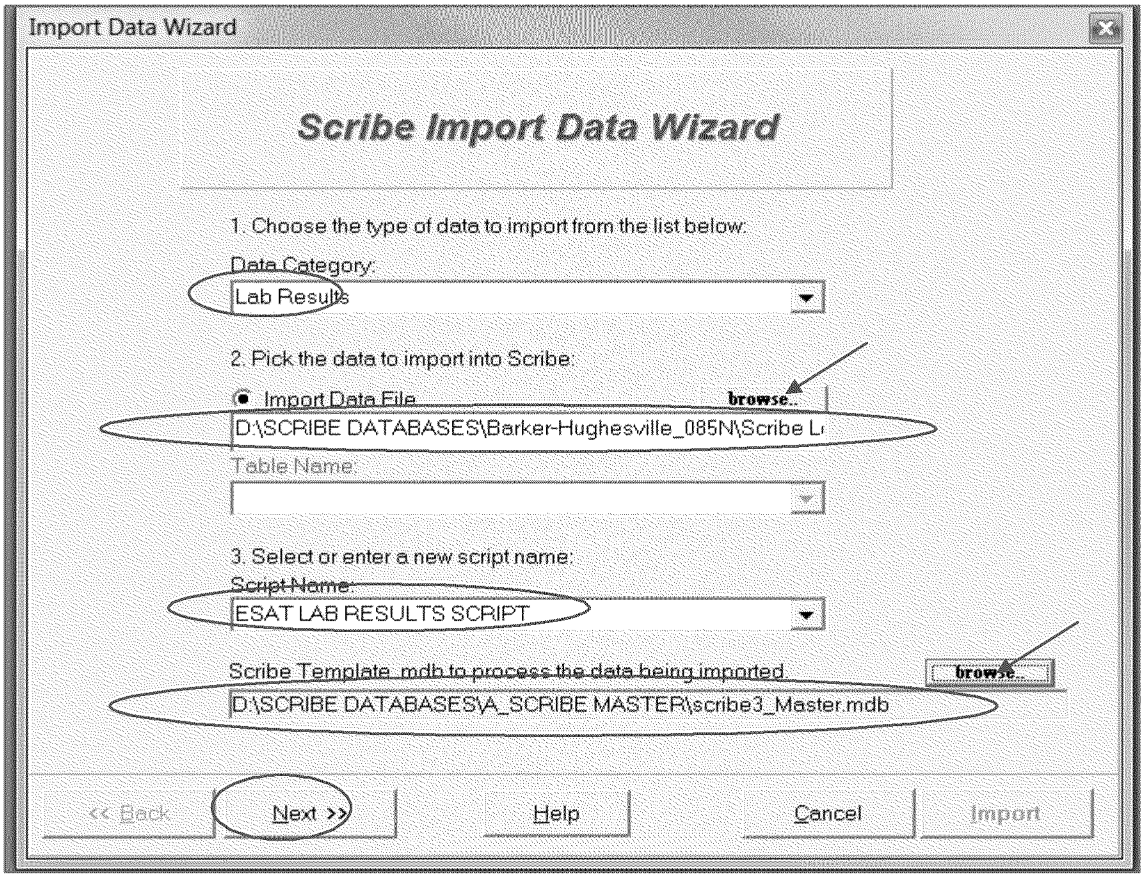


- Select "Lab Results" from the drop down arrow list in the Data Category field
- Select the correct EDD using the "browse" button above the Import Data File field

  Note: EDDs will be obtained from the folders located on the network drive pathways discussed in Section 5.1.

- Select "ESAT Lab Results Script" for the "Script Name" field
- Select the Master Scribe template using the "browse" button above the Scribe Template field; this template is located on the external drive in Scribe Databases/Master
- Select "Next"

Figure 5.7 Scribe "Import Data Wizard"

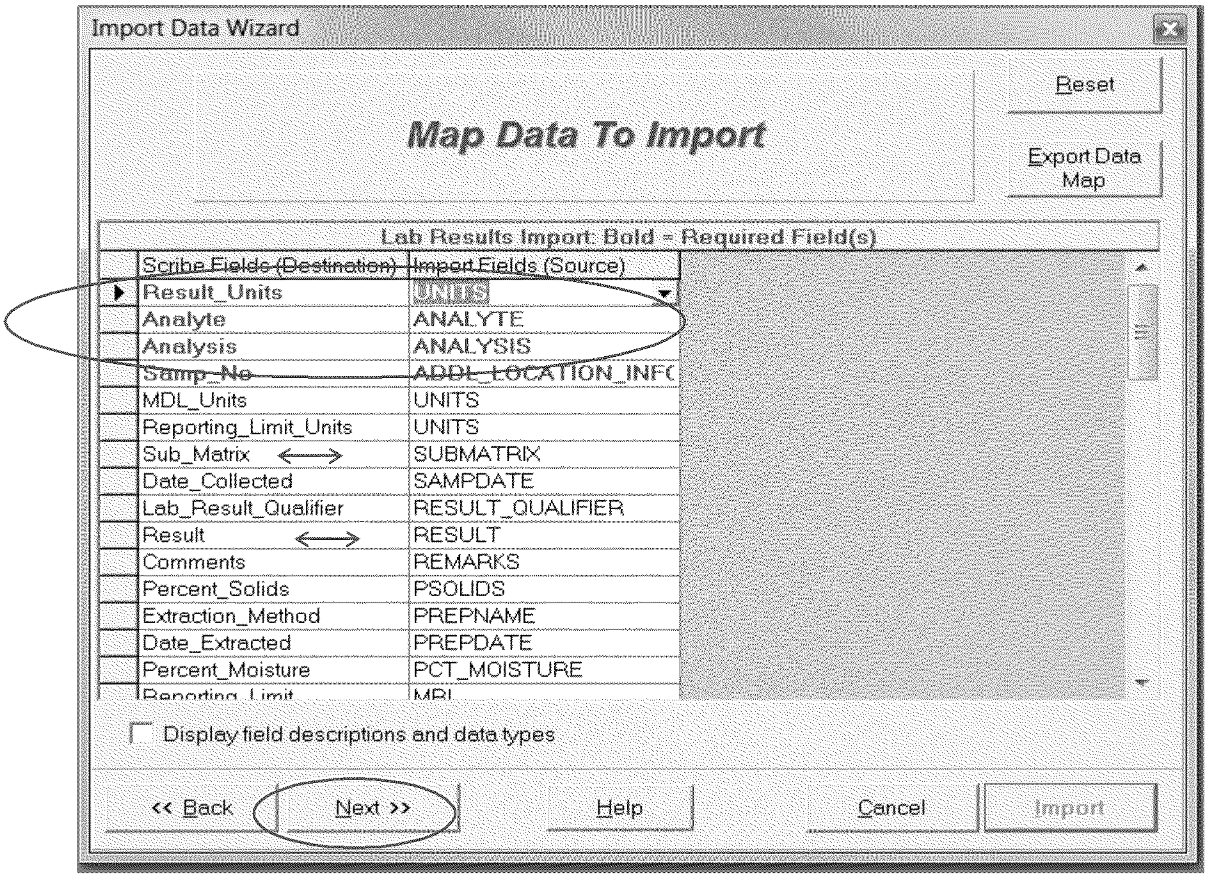


- Review the field mapping dialogue box to ensure that all fields match up with Scribe requirements

  Note: The fields in blue bolded text are required fields. The remaining fields (listed below the blue bolded font) may not match exactly in name. Ensure that they match in type and meaning regardless of the slight differences in names and case. In addition, not all "Import Fields" fields will be present for "Scribe Fields".

- Select "Next"

Figure 5.8 Scribe "Map Data to Import"

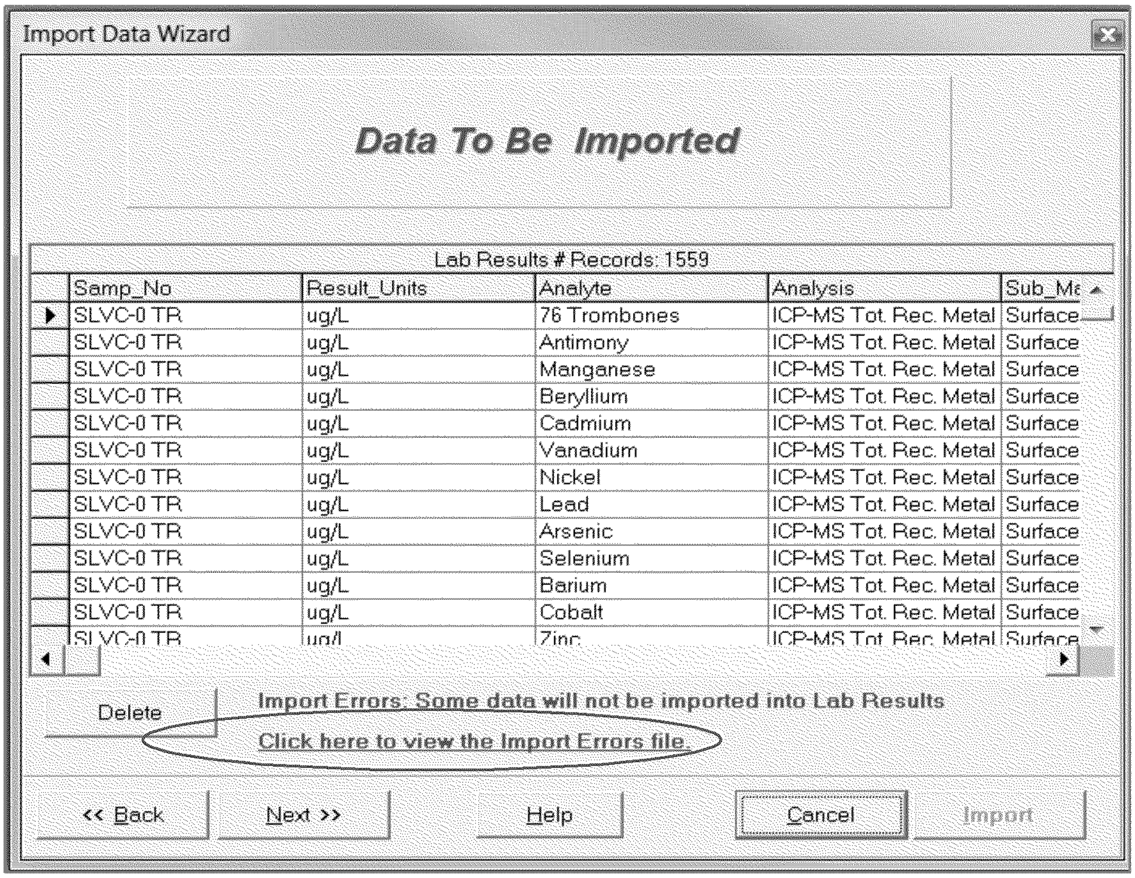


- Review the total in the "Lab Results # Records:" field
- Ensure that the total number of records in the "Lab Results # Records" field matches the total in the EDD

  Note: The Excel formatted EDD can be opened and reviewed to verify that the total number of samples to be imported matches the number of samples contained in the EDD.

- Select the blue bolded "Import Errors file" link to obtain a list of errors if the import is unsuccessful (list will open in Excel format)

Figure 5.9 Scribe "Data to be Imported" Import Errors

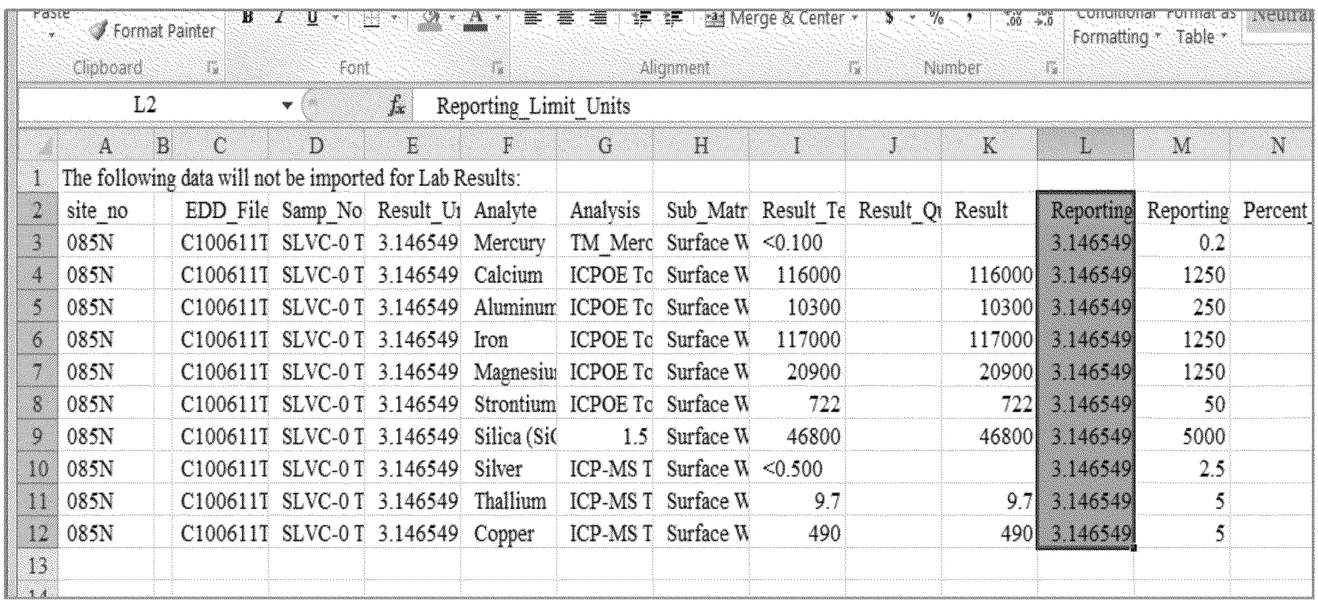


- Review the error report, open the appropriate EDD, and correct the errors. As shown in the example report below, the "Reporting Units" field has a numerical value, which is incorrect. Because this is a text field, the necessary correction would be to insert the appropriate unit text, which for this EDD would be ug/L.

  Note: The example below contains one of the common types of errors that may occur. That is, one that is correctable within the EDD itself and by the Scribe uploader. However, this is not the only possible error type. If a more complex error occurs, one that cannot be corrected within the EDD, an ESAT analyst may need to be contacted for assistance and the error may need to be corrected at an earlier point in the process.

  Errors within the Scribe program itself are essentially non-existent for the Analytical Support Group and Field Operations databases. If an upload error occurs, it is generally caused by incorrect selections of either the database script or the Scribe Master template. In those cases, return to the import screen and ensure that all scripts and templates selected are correct. If the database still shows errors that are not correctable in the EDD, the error(s) may be Scribe-related. If an error occurs that cannot be corrected within the EDD, or by correcting possible upload procedure errors, contact the Environmental Response Team (ERT) Software Support department for assistance. ERT can be contacted by email: ertsupport@epa.gov or by phone: 800-999-6990.

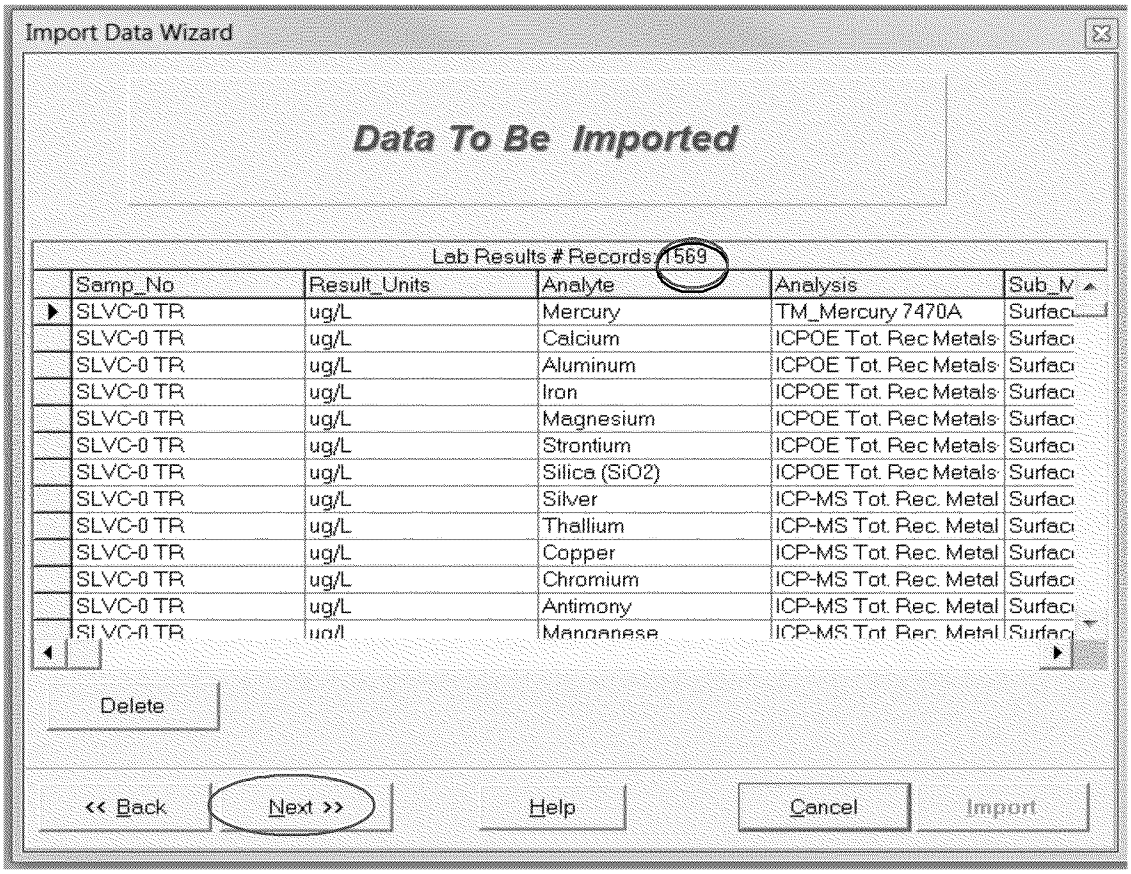
Figure 5.10 Scribe Example of Import Errors File



	A	B	C	D	E	F	G	H	I	J	K	L	M	N
1	The following data will not be imported for Lab Results:													
2	site_no		EDD_File	Samp_No	Result_Ui	Analyte	Analysis	Sub_Matr	Result_Te	Result_Qi	Result	Reporting	Reporting	Percent_
3	085N		C100611T	SLVC-0 T	3.146549	Mercury	TM_Merc	Surface W	<0.100			3.146549	0.2	
4	085N		C100611T	SLVC-0 T	3.146549	Calcium	ICPOE Tc	Surface W	116000		116000	3.146549	1250	
5	085N		C100611T	SLVC-0 T	3.146549	Aluminum	ICPOE Tc	Surface W	10300		10300	3.146549	250	
6	085N		C100611T	SLVC-0 T	3.146549	Iron	ICPOE Tc	Surface W	117000		117000	3.146549	1250	
7	085N		C100611T	SLVC-0 T	3.146549	Magnesiu	ICPOE Tc	Surface W	20900		20900	3.146549	1250	
8	085N		C100611T	SLVC-0 T	3.146549	Strontium	ICPOE Tc	Surface W	722		722	3.146549	50	
9	085N		C100611T	SLVC-0 T	3.146549	Silica (Si(	1.5	Surface W	46800		46800	3.146549	5000	
10	085N		C100611T	SLVC-0 T	3.146549	Silver	ICP-MS T	Surface W	<0.500			3.146549	2.5	
11	085N		C100611T	SLVC-0 T	3.146549	Thallium	ICP-MS T	Surface W	9.7		9.7	3.146549	5	
12	085N		C100611T	SLVC-0 T	3.146549	Copper	ICP-MS T	Surface W	490		490	3.146549	5	
13														

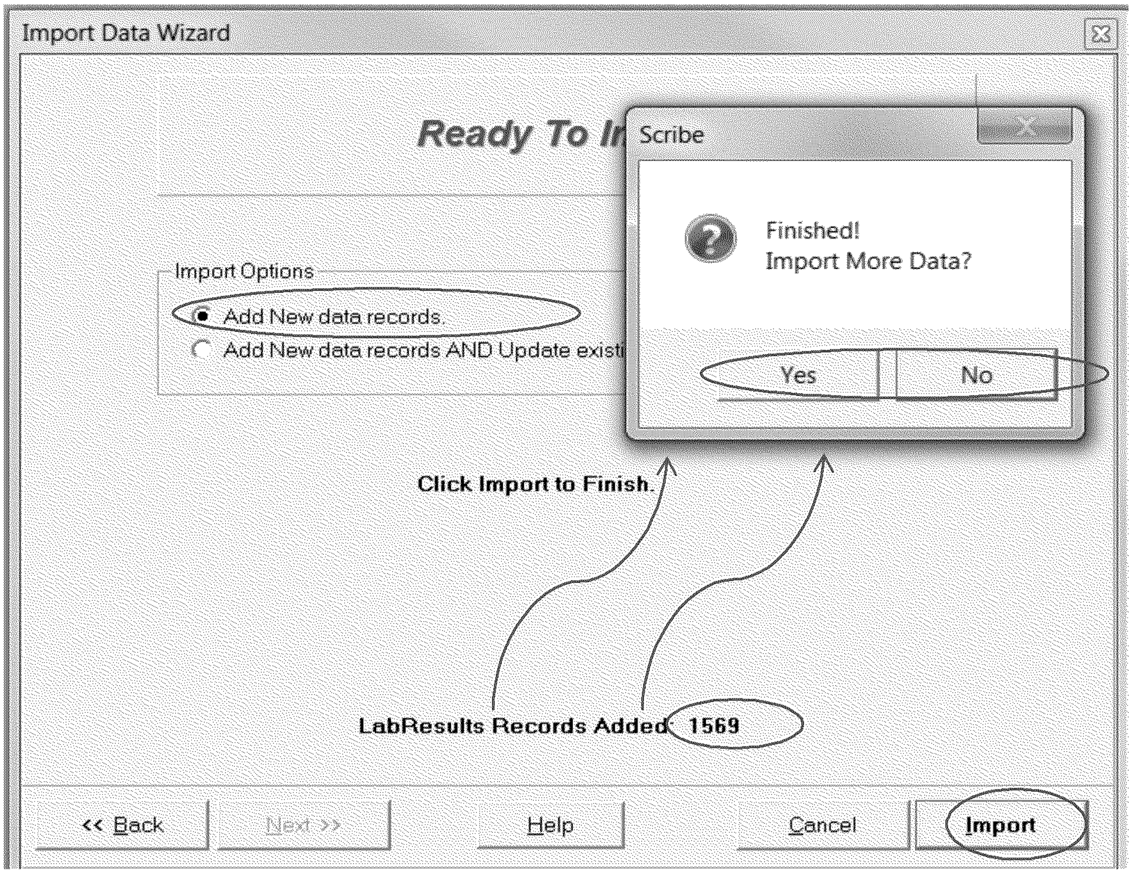
- Select "Next" once the errors have been corrected and continue the import process

Figure 5.11 Scribe Corrected "Data to be Imported"



- Select the "Add New Records" box
- Select "Import"
- Move the "Finished!" dialogue box away from the center portion of the "Import Data Wizard" dialogue box so that the "LabResults Records Added" total can be viewed
- Ensure that the "LabResults Records Added" is the same as the total in the "Data to be Imported" dialogue box (Figure 5.11)
- Select "Yes" if more EDDs for the same TO and Project will be loaded
- Select "No" if complete

Figure 5.12 Scribe "Import More Data?"



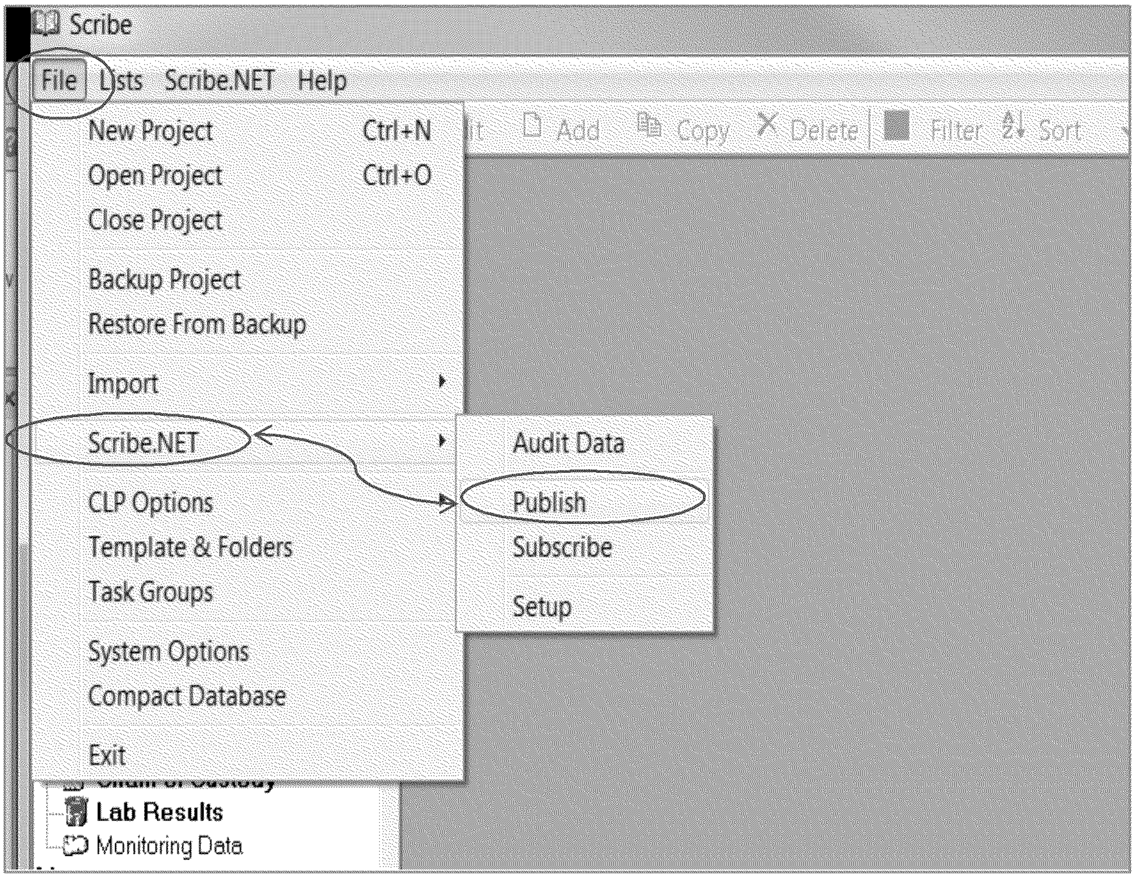
- Continue with all completed EDDs (those noted as ready to upload in the LIMS Tracking spreadsheet)

5.3 Publish Databases in Scribe

Once all EDD uploads for a specific project are loaded to Scribe, the project can be published to Scribe.NET.

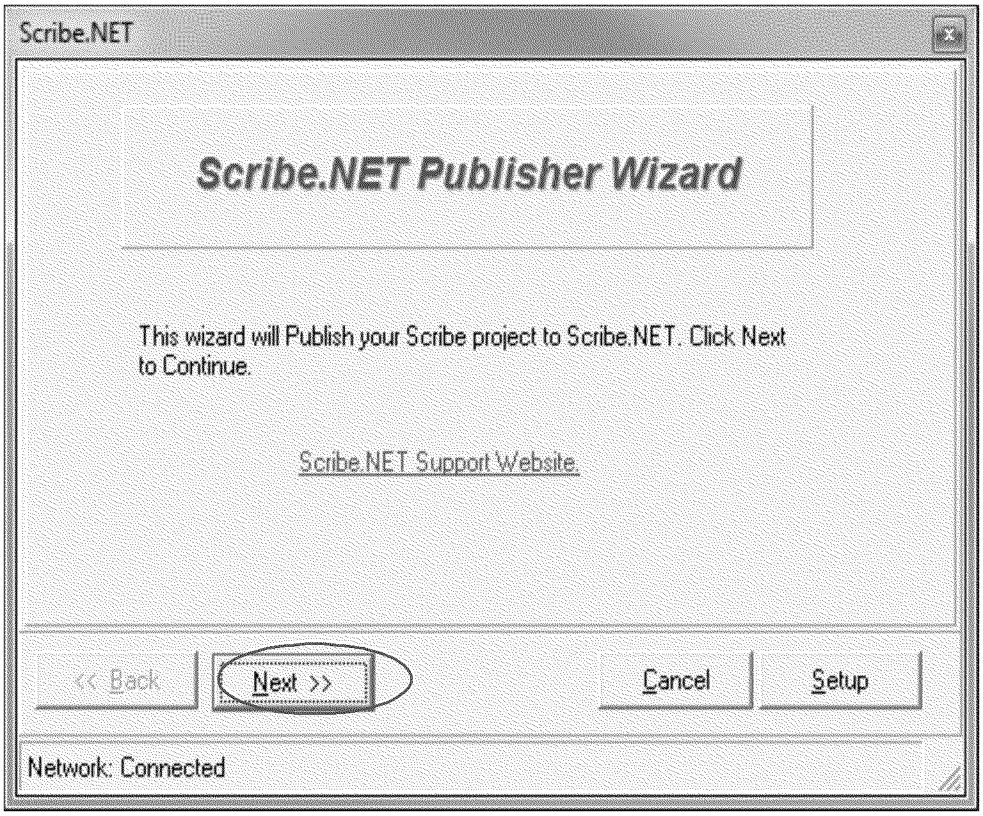
- Navigate to the LIMS Tracking spreadsheet on the network drive as described in Section 5.1 and shown in Figure 5.1
- Open the LIMS Tracking spreadsheet
- Follow the steps for opening a specific database as shown in Section 5.2, Figure 5.3, Figure 5.4, and Figure 5.5
- Select "File ->Scribe.net->Publish"

Figure 5.13 Scribe "File/Scribe.NET/Publish"



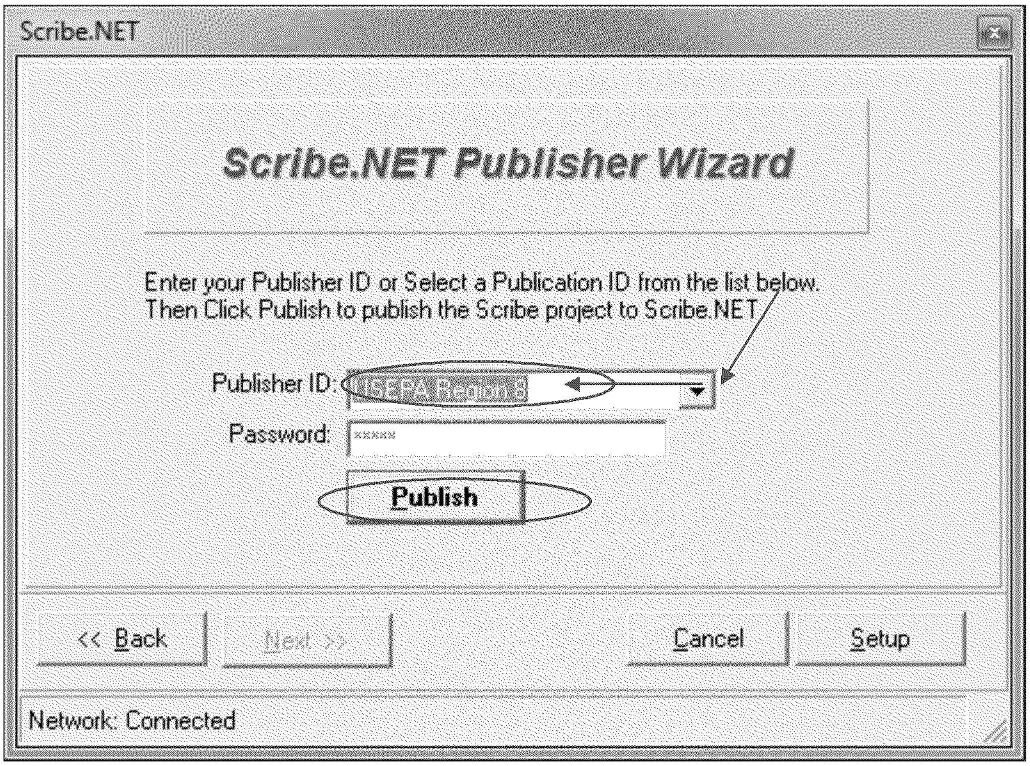
- Select "Next" on the Scribe.NET Publisher Wizard dialogue box

Figure 5.14 "Scribe.NET Publisher Wizard"



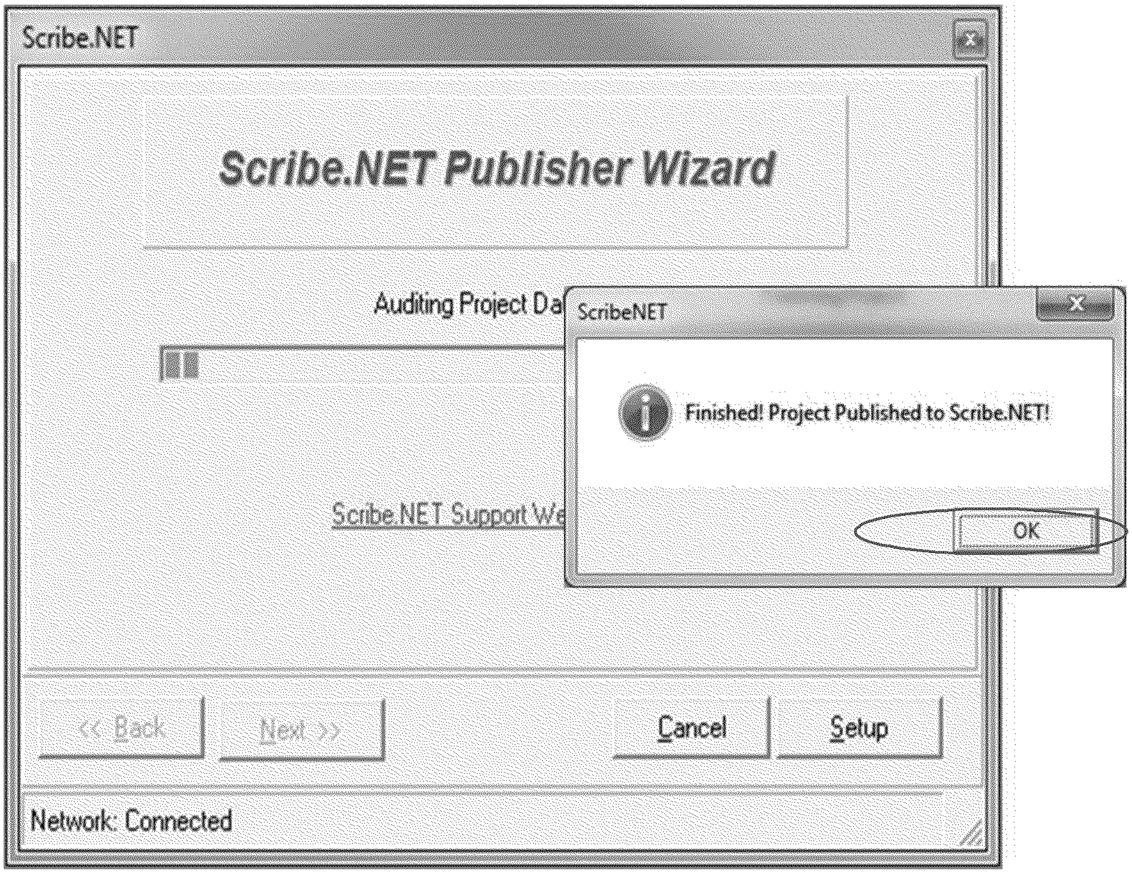
- Select "USEPA Region 8" from the dropdown list on the resulting dialogue box; leave the password field as is
- Select "Publish"

Figure 5.15 Scribe Publish Dropdown



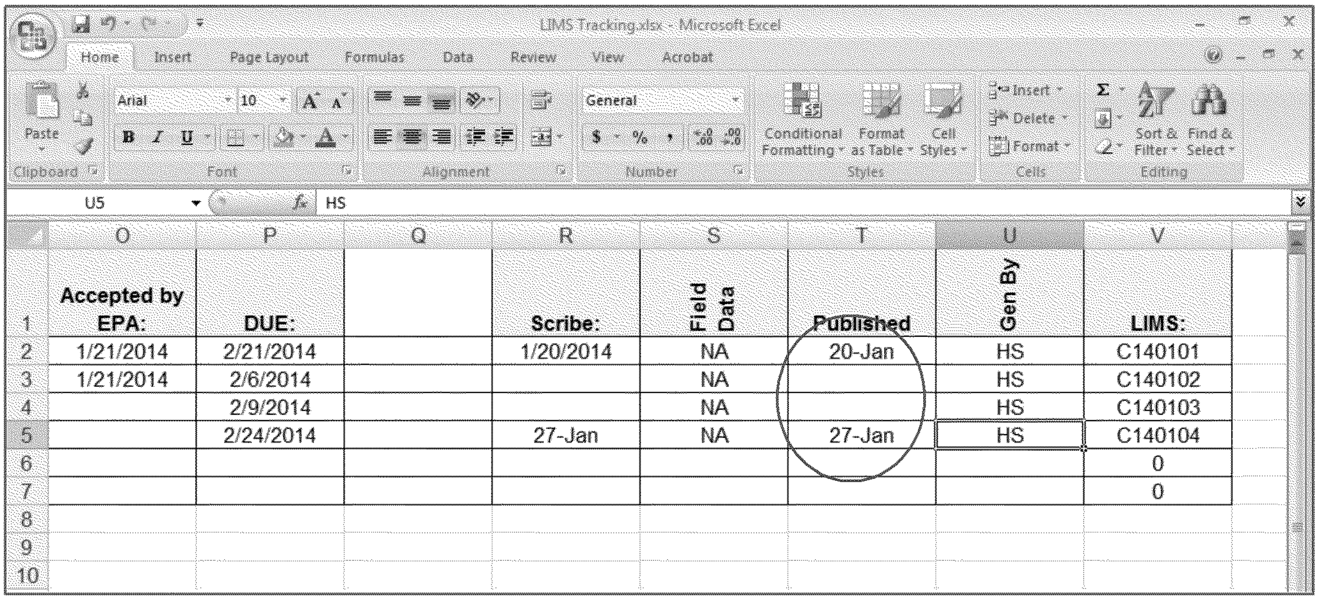
- Select "OK" in the "Finished! Project Published to Scribe.NET!" dialogue box

- **Figure 5.16 Scribe.NET "Project Published"**



- Move completed and published project folders to the correct TO subfolder titled "Final Folder" (located on the network drive, as described in Section 5.1)
- Open the LIMS Tracking spreadsheet and record the Publish date (date format defaults to spreadsheet formatting) for each Sample Event completed
- Continue with remaining TO Sample Events

Figure 5.17 LIMS Tracking Spreadsheet Input Published Date



	O	P	Q	R	S	T	U	V
1	Accepted by EPA:	DUE:		Scribe:	Field Data	Published	Gen By	LIMS:
2	1/21/2014	2/21/2014		1/20/2014	NA	20-Jan	HS	C140101
3	1/21/2014	2/6/2014			NA		HS	C140102
4		2/9/2014			NA		HS	C140103
5		2/24/2014		27-Jan	NA	27-Jan	HS	C140104
6								0
7								0
8								
9								
10								

6.0 Data Records and Management

The Data Package Coordinator will combine documents contained within each specific TO and Project folder and create and assemble the final Data Package for submittal to the client. Both the Excel and .csv versions of the EDD, as well as the data package and other associated documents, will be located in the appropriate TO Project file and Sample Event folder as shown in Figure 5.3.

7.0 Quality Control and Assurance

This SOP meets all the requirements of the ESAT Quality Management Plan.

8.0 References

ESAT Region 8, SOP,16-QAQ-03.00, Document Control, effective November 11, 2013.

ESAT Region 8, Quality Management Plan, version 7, effective June 2013.

TechLaw, Inc., Health and Safety Program Plan, effective November 2013.

TechLaw Inc., Corporate Quality Management Plan, effective November 2013.

United States Environmental Protection Agency, Environmental Response Team Software Support, accessed online at: http://www.ertsupport.org/scribe_home.htm. February 7, 2014.

United States Environmental Protection Agency Guidance for Preparing Standard Operation Procedures, EPA QA/G-6, April 2007.

Document Change History			
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Surface Water Sampling

APPROVED:

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This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to guide field personnel on general surface water sampling procedures. Not all situations are accounted for; therefore site reconnaissance is a key factor in determining sampling techniques that may be utilized. Always consult the site-specific Sampling and Analysis Plan and Quality Assurance Project Plan (SAP/QAPP) before deployment to a surface water sampling event.

2.0 SCOPE AND APPLICABILITY

This SOP is applicable to the collection of representative liquid samples from streams, rivers, lakes, ponds, lagoons, and surface impoundments utilizing direct method sampling procedures. This method may be varied or changed as required, dependent upon site conditions, equipment limitations or other procedural limitations. These are the preferred methodologies and should be implemented as closely as possible. Any deviations from these procedures should be discussed with site managers in order to confirm that data objectives are being met. All procedures employed should be documented and associated with the final report. Mention of trade names or commercial products does not constitute TechLaw, Inc. endorsement or recommendation for use.

This SOP is to be used in conjunction with other relevant and applicable documents which may include:

- Water Quality Measurement Procedures
- Sample Preservation Procedures
- Sediment Sampling Procedures
- Pore Water Sampling Procedures
- Field Sampling Procedures

The following documentation should be included to assist in preparing for and conducting surface water sampling activities:

- Health and Safety Plan (HASP)
- Sampling and Analysis Plan (SAP)
- Quality Assurance Project Plan (QAPP)
- Any other site-specific planning documents

3.0 SUMMARY OF METHOD

This SOP is intended to provide guidance on collection of surface water. Sampling situations vary widely, therefore, no universal sampling procedure can be recommended. However, sampling of aqueous liquids from the above mentioned sources is generally accomplished through use of the direct method technique (filling sample containers directly from the source) and transfer devices (collecting the sample with a container and then transferring to another container). This allows for the collection of representative samples of surface water from streams, creeks, rivers, lakes, ponds, and other impoundments. Note that for certain types of sampling, transfer devices are not appropriate. Volatile Organic Analysis and Semi-Volatile Organic Analysis samples should always be sampled directly if possible. Also, proper decontamination or conditioning of transfer devices must occur in order to avoid cross-contamination.

4.0 ACRONYMS AND DEFINITIONS

°C	Degrees Celsius
COC	Chain of Custody
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
EDI	Equal Discharge Increment
EWI	Equal Width Increment
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	High Density Polyethylene
LDPE	Low Density Polyethylene
mL	Milliliter
OSHA	Occupational Health and Safety Administration
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
VCF	Ventrical at Centroid of Flow

Equal Discharge Increment (EDI): A surface water sampling strategy that requires isokintetic water sampling from locations in a moving body of water that have the same discharge rate.

Equal Width Increment (EWI): A surface water sampling strategy that requires isokinetic water sampling from established equidistant intervals in a moving body of water.

Global Positioning System (GPS): A geospatial referencing tool that is used for mapping and identification.

Sampling and Analysis Plan (SAP): A site-specific document that details events to take place in the field.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

Quality Assurance Project Plan (QAPP): A site-specific document that specifies quality assurance activities and data quality objectives.

Ventrical at Centroid of Flow (VCF): A sampling strategy similar to EDI sampling where only one location along a transect is used to collected the isokinetic water sample.

5.0 HEALTH AND SAFETY

When working with potentially hazardous materials or in hazardous situations, personnel must understand and comply with the site-specific SAP/QAPP and HASP before the sampling event begins. More specifically, when sampling streams or surface impoundments containing known or suspected hazardous substances, adequate personal protective equipment such as nitrile gloves, safety glasses, and waders are necessary to prevent contact with contaminants during sampling. When entering a stream, hazardous situations may exist requiring the use of adequate personal safety equipment including personal floatation devices and non-slip footwear. When conducting sampling from a boat in an impoundment or flowing waters, appropriate boating safety procedures should be followed.

6.0 CAUTIONS

Only collect surface water samples if it can be done so safely. Many unsafe conditions exist on streams, rivers, ponds, and other surface water impoundments. Consult the site HASP before performing any sample collection.

7.0 INTERFERENCES

There are three primary interferences or potential problems with surface water sampling. These include cross-contamination of samples, improper sample collection, and improper sample preservation.

1. Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Refer to the Sampling Equipment Decontamination SOP #FLD-02.00.

2. Improper sample collection can involve disturbance of the stream substrate and/or sampling in an obviously disturbed area. To minimize potential for unrepresentative samples, consider sampling from downstream to upstream to avoid sampling where the substrate has been disturbed. Site sampling often involves multiple sampling procedures being deployed simultaneously. Therefore, well-organized team member coordination is essential to prevent improper sample collection.

3. Sample preservation is a critical component of sample collection. If the wrong preservative is used, the sample must be re-collected if possible. Improperly preserved samples cannot be analyzed. Refer to the Sample Preservation SOP #FLD-03.00 for proper field sample preservation guidelines.

8.0 PERSONNEL QUALIFICATIONS

Any personnel who are involved with field sampling activities must be cleared for health and safety. Clearance includes medical monitoring, respirator fit testing, and Occupational Health and Safety Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. Personnel who will be collecting surface water samples should familiarize themselves with this and other pertinent SOPs including: Sample Equipment Decontamination SOP #FLD-02.00; Sample Preservation SOP #FLD-03.00; Water Quality Measurements with the In-Situ® Multi-Parameter Meter SOP #FLD-09.00; Sample Custody and Labeling SOP #FLD-11.00; and General Field Sampling Protocols SOP #FLD-12.00.

9.0 EQUIPMENT AND SUPPLIES

Equipment needed for collection of surface water samples may include:

HASP Required Gear - personal floatation device, waders/gloves, proper footwear, safety glasses, insulating clothing for cold water, etc.

Mapping & Location Tools - GPS units, site/local area maps, compass, tape measure, survey stakes, pin flags, camera, 2-way radios

Documentation Supplies - field log book, field data sheet, Chains of Custody (COCs), labels, clear tape, pens, permanent marker, waterproof paper

Sampling Tools - plastic or other appropriate composition transfer device, bucket, rinse bottles, purified water, paper towels, filtering equipment, vacuum pump tool, vacuum pump stand, preservative, Ziploc™ plastic bags, cooler(s), ice, thermometer, preservative waste containment

Sample Containers - High-density polyethylene/Low Density Polyethylene (HDPE/LDPE) or other appropriate composition containers.

See Table 9.0-1 for a detailed list of surface water sampling equipment.

10.0 STANDARDS AND REAGENTS

Reagents will be utilized for preservation of samples and for decontamination of sampling equipment (refer to SOP's #FLD-02.00 and #FLD-03.00). The preservatives required are specified by the analysis to be performed and will be specified in the SAP/QAPP but usually include nitric acid (Total Recoverable and Dissolved Metals samples) and phosphoric acid (Dissolved and Total Organic Carbon samples). Field sampling personnel should also be aware of any special sampling considerations, contamination issues, and sample compositing and mixing methods that could affect the sampling efforts. Appropriate regional guidance and procedures should be consulted for detailed sample collection, preservation, handling and storing, equipment decontamination, and Quality Assurance/Quality Control (QA/QC) procedures. Field sampling personnel should preserve and immediately cool all water samples to 4 degrees Celcius (°C) (±2°C) upon collection and samples should remain <6°C until the time of analysis (do not freeze water samples).

11.0 PROCEDURES

11.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods required, and the types and amounts of equipment and supplies needed. Use the site-specific SAP/QAPP for guidance to determine which kind of samples need to be collected.

2. Obtain the necessary sampling and monitoring equipment and ensure it is in working order.

3. Decontaminate equipment according to the procedures outlined in SOP #FLD-02.00, or use triple rinsed, dedicated disposable sample containers (non-filtering and not pre-

preserved).

4. Prepare scheduling and coordinate with staff, clients, and regulatory agencies where appropriate. It is also important to obtain site access agreements if sampling is to occur on private property.

5. Perform a general site survey prior to site entry in accordance with the site-specific HASP.

6. Use stakes, flagging, GPS markers, or photos to identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

Generally, factors to consider in the selection of a device for sampling liquids in streams, rivers, lakes, ponds, lagoons, and surface impoundments are:

a. Can the sample be collected directly from the source? (i.e. is a transfer device needed for sample collection?)

b. What is the desired depth at which you wish to collect the sample?

c. What is the overall depth and flow direction of river or stream?

d. What type of analysis will be run (total recoverable metals, dissolved metals, alkalinity and anions, etc.)?

11.2 Sample Container Composition

A sample container should be selected based on analysis to be performed. A triple rinse should be performed on any sample container being used for direct sample collection. Filtered sample bottles or pre-preserved bottles should never be rinsed first.

11.3 Sample Collection

1. Direct collection is the optimal procedure for sample collection. After rinsing, the sample should be collected in a well-mixed area, as close to the middle of the stream as possible, in-between the streambed and the surface. The sample should be capped immediately.

2. Preserve the sample if appropriate. Refer to the site-specific SAP and SOP #FLD-03.00 for correct methods. The sample should be clearly labeled (and bagged in order to keep samples from the same location together) before being placed in a cooler on ice.

3. Record all pertinent site data (usually date, time, pH, conductance, dissolved oxygen, temperature, site ID, and anomalies) in the field logbook, field data sheets and/or sample container labels.

4. Complete the COC record. Refer to SOP #FLD-11.00 for guidelines on sample

custody and labeling documentation.

5. Attach custody seals to cooler prior to shipment where applicable. Refer to SOP #FLD-11.00 for guidelines on sample custody and labeling.

6. If a non-direct method was used for sampling (i.e. a bucket from a bridge or a dip sampler), decontaminate or condition all sampling equipment prior to the collection of additional samples with that sampling device as required by SOP #FLD 02.00. Sections 11.3.4 and 11.3.5 describe a few of the non-direct sampling methods that may be useful in Region 8.

7. If sampling on private property, sample receipts will be provided to property owners for all samples taken and removed from the property.

11.3.1 Direct Method

For streams, rivers, and lakes, the direct method may be utilized to collect water samples from the surface directly into the sample container. For shallow stream stations, collect the sample under the water surface while pointing the sample container upstream; the container must be upstream of the collector. Samples should be collected prior to all other activities as specified in the SAP/QAPP to avoid disturbing the substrate. Rinse the sample container three times (unless it's filtered or pre-preserved) with site water before procuring a sample. When using the direct method, do not use pre-preserved sample bottles as the collection method may dilute the concentration of preservative necessary for proper sample preservation.

11.3.2 Dip Sampling

Dip sampling is useful in situations where a sample is to be recovered from an outfall pipe or from a bridge where direct access is limited. The long handle or rope on such a device allows access from a discrete location. Sampling procedures are as follows:

1. Assemble the device in accordance with the manufacturer's instructions.

2. Extend the device to the sample location and collect the sample by dipping the sampler into the water at the sampling location.

3. Triple rinse the sampler with the site water

4. Retrieve the sampler and transfer the sample to the appropriate (triple rinsed) sample container

11.3.3 Synoptic Sampling

Synoptic sampling is a strategy used for evaluating a surge of water as it moves downstream. In general, the flow rate should be determined before executing a synoptic sampling event. Flow measurements are used to calculate when a surge of water will pass by a certain point or sample location. Floating visual objects may also be used to accurately determine when a surge of water passes by a sampling location. The synoptic sampling strategy is useful in determining where contaminants may be entering a watershed through seeps, fens, or other inflows that may not be visible. Below is a general guideline on synoptic sampling:

- Identify sample locations
- Conduct flow measurements; or test a floating object (ping pong ball, tangerine, etc.) to see if it will float through to the last sampling location (some objects may get caught in eddies or shallow areas)
- Calculate when the surge of water will pass through each sampling location
- Position sampling personnel or equipment where a sample can be captured at the required time
- Process samples

11.3.4 Large Stream or River Sampling

There are several techniques for sampling large rivers or streams. The most commonly used are Equal-Width Increment (EWI) sampling, Equal Discharge Increment (EDI) sampling, Single Vertical at Centroid of Flow sampling (VCF), dip sampling (section 11.3.2), direct method sampling (section 11.3.1), discrete sampling, and pump sampling (USGS, 2006). Below is a brief description of the sampling methods that have not yet been mentioned in this document:

EWI Method – A stream cross-section is divided into equal width intervals, and samples are collected by lowering and raising a sampler through the water column at the center of each interval. This produces a discharge weighted sample that is proportional to stream flow. This method cannot be used if the stream flow is less than what is required to fill the sampler during the isokintetic (constant rate) motion.

EDI Method – The objective of this method is to obtain a discharge weighted sample that represents the entire flow through a cross-section by obtaining a series of samples. For this method, the flow in the cross-section must be divided into points of equal discharge. Equal volume and depth integrated samples are collected at the center of the equal discharge interval along the cross-section. Flow measurements or historical data is necessary to determine interval number (usually more than 4, but less than 20) and location. This method may also require additional personnel to sample a large cross-section. If conducted properly, both the EWI and EDI methods should produce identical results.

VCF Method – This method is a simple version of the EDI method, but only one sample is collected at flow center (usually a smaller river or stream). This is to be used if the sample location is known to be homogenous and is warranted by the sampling plan objectives. Flow data should be obtained to determine where the flow center is located.

11.3.5 Shallow Stream and Still Water Sampling

Shallow streams and still water such as a pond are common locations to sample in Region 8. Below are three methods (in addition to ones mentioned above) that may be required.

Discrete (point) Sampling – This method is achieved by lowering a sample container to a specific depth in a body of water then opening and closing the container to obtain the sample.
Pump Sampling – This method is used to collect single point samples using a suction-lift or submersible pump. These are not used for collecting isokinetic samples (EWI, EDI, or VCF). Using the pump method is limited by electrical needs in remote areas. Always consult the SAP for sampling objective requirements.

Syringe Sampling – A 50 mL syringe can be used to collect sample from very shallow locations to avoid contact with substrate.

12.0 DATA RECORDS AND MANAGEMENT

Once collected, samples are preserved, labeled, and stored for transport. A COC must accompany all samples during transport and transfer between entities. Sample labels should contain the following information:

- Site Identification
- Date sampled
- Sampler initials
- Time
- Analysis to be performed

13.0 QUALITY CONTROL AND ASSURANCE

1. The following general QA procedures apply:

2. All data must be documented on field data sheets or within site logbooks.

3. In general, concurrent (duplicate) sample collection at a frequency of 10% is required for most sampling activities. Blanks at a frequency of one per day are also generally required. Consult the corresponding SAP/QAPP for specific QA/QC sampling frequency. Below is a list of typical QA/QC sample types and the inaccuracy they are intended to detect:

   - Field blank – checks cross-contamination during sample collection, preservation, and shipment as well as in the laboratory.
   - Equipment blank – equipment contamination due to inadequate decontamination procedures
   - Temperature blank – provides an accurate temperature measurement of field samples upon arrival to the laboratory and establishes whether the temperature range has been maintained while in transit
   - Trip blank – checks contamination of samples during handling, storage, and shipment from the field to the laboratory; carried through the same sampling and handling

protocols as field samples and placed in the cooler for the duration of the trip.

- MS/MSD – checks accuracy and precision of organic or inorganic analyses in specific sample matrices. They are collected from areas know or suspected to be contaminated.
- Sequential replicates – samples are pulled one after another to detect variability among field activities (collection, preservation, handling, etc.)
- Split samples – division of one sample into two, then submitting for identical analysis in order to detect variability in the process from collection to analysis
- Concurrent or collocated replicate samples (often referred to as “duplicate” samples) – two samples collected at the same location at the same time, intended to detect variability inherent in collection, processing, and handling procedures; Relative percent difference is usually calculated from these samples.

4. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer unless otherwise specified in the work plan or SAP/QAPP. Equipment calibration activities must be conducted and documented prior to sampling and/or operation of equipment.
5. Document any deviations from SOPs, work plan, SAP/QAPP, etc.

14.0 REFERENCES

Environmental Protection Agency (EPA) Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

U.S. Geological Survey. 1977. National Handbook of Recommended Methods for Water Data Acquisition. Office of Water Data Coordination, Reston, Virginia. (chapter updates available)

U.S. Geological Survey 2006. National Field Manual for the Collection of Water Quality Data. Chapter A4, version 2.0, Office of Water Quality.

Environmental Protection Agency (EPA), 1984. Characterization of Hazardous Waste Sites - A Methods Manual: Volume II. Available Sampling Methods, Second Edition. EPA/600/4-84-076.

Environmental Protection Agency (EPA), Environmental Response Team, Standard Operating Procedures - Surface Water Sampling: U.S. Environmental Protection Agency, 1994, SOP #2013.

Table 9.0-1 Surface Water Sampling Equipment

Category	Item	Use	Comment
Health and Safety	Gloves	Protection from absorption of contaminants	Nitrile or neoprene are recommended
Health and Safety	Waders	Slip/contaminant protection, warmth	Any type are acceptable
Health and Safety	Safety Glasses	Eye protection	Sunglasses for UV protection
Health and Safety	Layered Clothing	Protection from hypothermia	Polyester base layers only
Mapping/Location	GPS unit	Sample station locating	Pre-loaded with site locations
Mapping/Location	Maps	Location identification	Most current information required
Mapping/Location	Two-way radios	Communication	Extra batteries or charger required
Documentation	Field Logbook	Site data and conditions documentation	Waterproof pages
Documentation	Field Data Sheets (waterproof)	Marsh McBirney Flows	
Documentation	Chain of Custody	Sample handling/identification	Generated using Scribe
Documentation	Labels	Sample identification	Generated using Scribe
Documentation	Clear tape & Scissors	Label protection	
Sampling Tools	Bucket/Transfer Device	Sample transfer (if required)	Decontaminated between samples
Sampling Tools	Vacuum Pump	Dissolved sample processing	
Sampling Tools	Vacuum Stand	Dissolved sample processing	
Sampling Tools	Ziploc baggies	Sample containment	
Sampling Tools	Water Chemistry meters	In-situ water quality data gathering	Temp, pH, dissolved oxygen, conductivity
Sampling Tools	Flow measurement equipment	Flow measurements	
Sampling Tools	Cooler	Sample containment	Samples to be kept at 4°C
Sampling Containers	250 ml HDPE/LDPE	Total recoverable metals samples	
Sampling Containers	250 ml HDPE/DLPE filtered bottles	Dissolved Metals/ DOC samples	Single use only
Sampling Containers	500 ml HDPE/LDPE	Alkalinity+Anions samples	
Sampling Containers	VOA Vials	Volatile and Semi-Volatile organics analysis	
Reagents	Nitric Acid(HNO3) ampules	For preserving metals samples	
Reagents	Phosphoric Acid (H3PO4) ampules	For preserving DOC samples	
Reagents	Hydrochloric (HCl) Acid	For preserving VOA samples	
Reagents	CaCO3 Acid waste Containment	Ampule/acid waste disposal	Usually in a 1 liter cubitainer
Reagents	pH and Conductivity standards	Calibration of water quality equipment	Quantity for each day of sampling

Shallow Stream Sediment Sampling

APPROVED:

ESAT Region 8 QA Coordinator	Date 06/06/12
ESAT Region 8 Team Manager	Date 6/6/12
EPA Task Order Project Officer	Date 7/10/12
ESAT Region 8 Task Lead	Date 6/8/12

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide field personnel a set of guidelines for the proper collection of stream sediment samples.

2.0 SCOPE AND APPLICABILITY

This SOP is applicable to the collection of shallow stream sediment samples. Analysis of sediment may be biological, chemical, or physical in nature and may be used to determine the following:

- Toxicity
- Biological availability and effects of contaminants
- Benthic biota
- Extent and magnitude of contamination
- Contaminant migration pathways and source
- Fate of contaminants
- Grain size distribution

The methodologies discussed in this SOP are applicable to the sampling of sediment in lotic environments. They are generic in nature and may be modified in whole or part to meet the handling and analytical requirements of the contaminants of concern, as well as the constraints presented by site conditions, equipment limitations and requirements of the site-specific Sampling and Analysis Plan (SAP). However, if modifications occur, they should be documented in field data sheet/field notebook and discussed in reports summarizing field activities and analytical results. For the purposes of this procedure, sediments are those mineral and organic materials situated beneath an aqueous layer in rivers and streams. Mention of trade names or commercial products for use in sediment sample collection does not constitute endorsement or recommendation for use.

3.0 SUMMARY OF METHOD

Sediment samples may be collected using a variety of methods and equipment, depending on the depth of the aqueous layer, the portion of the sediment profile required (surface vs. subsurface), the type of sample required (disturbed vs. undisturbed), contaminants present, and sediment type. Sediment is collected from beneath an aqueous layer directly, using a hand held device such as a shovel, trowel, or plastic scoop. Following collection, sediment is transferred from the sampling device to an appropriate sample container. If composite sampling techniques are employed, multiple grabs are placed into a container constructed of inert material, homogenized, and transferred to sample containers appropriate for the analyses requested.

The homogenization procedure should not be used if sample analysis includes volatile organics. In this case, if sediment is to be analyzed for volatile organics then the sample must be transferred to the appropriate sample container directly after collection. The sample bottle is filled completely and tapped lightly to get the trapped air out of the bottle. If the sediment settles in the bottle creating airspace then additional sediment should be collected. Repeat this step as many times necessary in order to have the sample bottle completely filled without having any air gaps.

4.0 ACRONYMS AND DEFINITIONS

COC	Chain of Custody
GPS	Global Positioning Systems
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	High-Density Polyethylene
OSHA	Occupational Health and Safety
QA	Quality Assurance
SAP/QAPP	Sampling and Analysis Plan/Quality Assurance Project Plan
SOP	Standard Operating Procedure

Chain of Custody (COC): A chronological document that tracks movement of samples between entities from collection to disposal.

Composite Sampling: Sampling from several points or intervals and consolidating them into a larger sample.

Discrete Sampling: Sampling from a single location.

Global Positioning System (GPS): A geospatial referencing tool that is used for mapping and identification

Health and Safety Plan (HASP): A site specific document that identifies safety hazards and proper safety procedures. This normally includes hospital route maps and material safety data sheets.

Sampling and Analysis Plan (SAP): A site specific document that specifies events to take place in the field.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

Quality Assurance Project Plan (QAPP): A site specific document that specifies quality assurance activities and data quality objectives.

5.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow United States Environmental Protection Agency (EPA), Occupational Safety and Health Agency (OSHA), and corporate health and safety procedures. More specifically, when sampling sediment from water bodies, physical hazards must be identified and adequate precautions must be taken to ensure the safety of the sampling team. The team member collecting the sample should not get too close to the edge of the water body, where bank failure may cause loss of balance. To prevent this, the person performing the sampling should be on a lifeline, and be wearing adequate protective equipment. If sampling from a vessel, appropriate protective measures and procedures must be implemented.

6.0 CAUTIONS

Only collect sediment samples if it can be done so safely. Many unsafe conditions exist on streams and rivers. Also, review the SAP/QAPP or any other planning documents for analytical requirements and equipment selection. Consult the site HASP before performing any sample collection.

7.0 INTERFERENCES

Substrate particle size and organic matter content are a direct consequence of the flow characteristics of a water body. Contaminants are more likely to be concentrated in sediments typified by fine particle size and high organic matter content. This type of sediment is most likely to be collected from depositional zones. In contrast, coarse sediments with low organic matter content do not typically concentrate pollutants and are generally found in erosion zones.

8.0 PERSONNEL QUALIFICATIONS

All personnel who participate in field activities are required to obtain clearance in three mandatory health and safety programs: medical monitoring, respirator fit testing, and Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. In addition to this, any personnel who will participate in sediment sampling activities must read, understand, and sign the site specific HASP and the associated SAP/QAPP. Additionally, field personnel would benefit from understanding relevant SOPs including Sampling Equipment Decontamination SOP FLD 02.00, Sample and Labeling SOP FLD 11.00, and the General Field Sampling Protocols SOP FLD 12.00.

9.0 EQUIPMENT AND SUPPLIES

Equipment needed for collection of sediment samples may include:

> Health and Safety Plan (HASP) - Personal floatation device, life line, neoprene waders/gloves, proper footwear, safety glasses, insulating clothing for cold water, etc.
>
> Mapping & Location Tools - GPS units, site/local area maps, tape measure, compass, survey stakes, pin flags, camera, and 2-way radios.
>
> Documentation - Field log book, field data sheet, chain of custody (COC), labels & clear tape, pens/sharpie, waterproof paper.
>
> Sampling Tools - Plastic or other appropriate composition scoop, shovel, spade, trowel, homogenization container with mixing tool, rinse bottle, purified water, and paper towels.
>
> Sample Containers -High-density polyethylene (HDPE) or other appropriate composition containers (50 mL and 1 liter [bulk] are frequently used), amber glass jars (organics analysis) labels, clear tape, pens, permanent marker, sealable plastic bags, cooler(s), and ice.

See Table 9.0-1 for a detailed list of sediment sampling equipment.

10.0 STANDARDS AND REAGENTS

Reagents are not used for preservation of sediment samples.

11.0 PROCEDURES

11.1 Sample Preservation, Containers, Handling and Storage

Chemical preservation of solids is not recommended. Cooling to 4 degrees Celsius (ºC) is recommended for sediment samples. HDPE containers with Teflon™ lined caps are typically used for sediment samples. Sample container size is typically 50 milliliter (mL) for metal analysis and 1 liter for sediment toxicity testing. However, the sample volume is a function of the analytical requirements and will be specified in the SAP/QAPP. If analysis of sediment from a discrete depth or location is desired, sediment is transferred directly from the sampling device to a labeled sample container(s) of appropriate size and construction for the analyses requested. Transfer is accomplished with a decontaminated stainless steel or plastic lab spoon or equivalent.

If composite sampling techniques or multiple grabs are employed, equal portions of sediment from each location are deposited into a stainless steel, plastic, or other appropriate composition containers. The sediment is then homogenized thoroughly, to obtain a composite sample that is representative of the area and is then transferred to a labeled container. Transfer of sediment is accomplished with a stainless steel or plastic lab scoop or equivalent. Samples for volatile organic analysis must be transferred directly from the sample collection. It is important that when collecting sediment for volatile organic compounds analysis, the sample container is filled completely full and the sample container is tapped lightly to ensure all air is purged from the sample. This is done to minimize loss of contaminant due to volatilization.

All sampling devices should be decontaminated following procedures described in the Sample Equipment Decontamination SOP FLD 02.00. The sampling device should remain in its wrapping until it is needed. Each sampling device should be used for only one sample. Although disposable sampling devices for sediment are generally impractical due to cost and the large number of sediment samples which may be required, such devices may prove efficient and effective for difficult terrain/remote locations. Sampling devices should be cleaned in the field using the decontamination procedure described in the Sampling Equipment Decontamination SOP FLD 02.00.

11.2 Preparation

Determine the objective(s) and extent of the sampling effort. Obtain access to private property if sample locations are located within private boundaries. The sampling methods to be employed, and the types and amounts of equipment and supplies required will be a function of site characteristics and objectives specified in the SAP and QAPP.

- Obtain the necessary sampling and monitoring equipment.
- Prepare schedules, and coordinate with staff, client, and regulatory agencies where appropriate.
- Decontaminate or pre-clean equipment, and ensure that it is in working order.

- Perform a general site survey prior to site entry in accordance with the site-specific HASP.
- Use stakes, flagging, or buoys in addition to using a GPS (Refer to SOP FLD 07.00) to identify and mark all sampling locations. Specific site factors including flow regime, basin morphology, sediment characteristics, depth of overlying aqueous layer, contaminant source, and extent and nature of contamination should be considered when selecting sample locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

11.3 Sample Collection

Selection of a sampling device is most often contingent upon the depth of water at the sampling location and the physical characteristics of the sediment to be sampled. The following procedure consists of sampling surface sediment with a scoop, trowel or shovel from beneath a shallow aqueous layer:

For the purpose of this method, surface sediment is considered to range from 0 to 1 inch in depth and a shallow aqueous layer is considered to range from 0 to 12 inches in depth. Collection of surface sediment from beneath a shallow aqueous layer can be accomplished with tools such as spades, shovels, trowels, and scoops. Although this method can be used to collect both unconsolidated/consolidated sediment, it is limited somewhat by the depth and movement of the aqueous layer. Deep and rapidly flowing water may render this method less accurate than other methods such as utilizing a handheld dredge or coring device. However, representative samples can be collected with this procedure in shallow sluggish water provided care is demonstrated by the sample team lead. A plastic scoop will suffice in most applications. Care should be exercised to avoid the use of devices plated with chrome or other materials.

The following procedure will be used to collect sediment with a scoop, shovel, or trowel:

1. Using a decontaminated sampling implement, collect the desired thickness and volume of sediment from the sampling area.

2. Transfer the sample into an appropriate sample or homogenization container. Ensure that non-dedicated containers have been adequately decontaminated.

3. Surface water should be decanted from the sample or homogenization container prior to sealing or transfer; care should be taken to retain the fine sediment fraction during this procedure.

11.3.1 Composite Sampling

Composite sampling consists of taking several sub-samples from a location and consolidating them into a larger sample. If data quality objectives dictate that each sub-sample of a composite be measured, it can be done two ways; by mass or by volume. For remote site field sampling activities (such as ones that typically occur in Region 8), it is recommended that sub-samples be measured by volume. This can be done with a graduated beaker/measuring cup or cylinder. Place the sub-sample in the measuring device, record the measurement, and transfer the sub-sample into a larger container where the complete composite sample will be processed.

Composite representative sample collection can also be accomplished without measurement of sub-samples. For sediment collection that does not require sub-sample measurement, larger amounts of sample can be collected in areas where sediment is more readily available. This method is used very frequently in high-gradient streams such as those found in the region.

11.3.2 Discrete Sampling

Discrete sampling consists of taking a sample from a single location. This method requires that the selected location for a sediment sample have sufficient amount of material for the analytical requirements. In general, sediment samples in a stream are difficult to obtain from a single location; therefore composite samples are more commonly collected.

12.0 DATA RECORDS AND MANAGEMENT

Once collected, samples are labeled and stored for transport (at 4°C). A COC must accompany all samples during transport and transfer between entities. Sample labels should contain the following information:

- Site Identification
- Date sampled
- Sampler initials
- Time
- Analysis to be performed

13.0 QUALITY CONTROL AND ASSURANCE

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the SAP and QAPP. Equipment inspection must occur prior to sampling, and they must be documented.

3. QA samples should be sampled at a standardized frequency. Field duplicates are generally sampled at a rate of 1:20.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

Mason, B.J., Preparation of Soil Sampling Protocol: Technique and Strategies. 1983 EPA-600/4-83-020.

Barth, D.S. and B.J. Mason, Soil Sampling Quality Assurance User's Guide. 1984 EPA-600/4-84-043.

U.S. EPA. Characterization of Hazardous Waste Sites - A Methods Manual: Volume II. Available Sampling Methods, Second Edition. 1984 EPA600/4-84-076.

de Vera, E.R., B.P. Simmons, R.D. Stephen, and D.L. Storm. Samplers and Sampling Procedures for Hazardous Waste Streams. 1980 EPA-600/2-80-018.

U.S. EPA, Environmental Response Team, standard operating procedures—Sediment Sampling: U.S. Environmental Protection Agency, 1994, SOP #2016.

Table 9.0-1: Sediment Sampling Equipment

Category	Item	Use	Comment
Health and Safety	Gloves	Protection from absorption of contaminants	Nitrile or neoprene are recommended
Health and Safety	Waders	Slip/contaminant protection	Any type are acceptable
Health and Safety	Safety Glasses	Eye protection	Sunglasses for UV protection
Health and Safety	Layered Clothing	Protection from hypothermia	Polyester base layers only
Mapping/Location	GPS unit	Sample station locating	Pre-loaded with site locations
Mapping/Location	Maps	Location identification	Must contain most current information
Mapping/Location	Two-way radios	Communication	Extra batteries or charger required
Documentation	Field Logbook	Site data and conditions documentation	Waterproof pages
Documentation	Chain of Custody	Sample handling/identification	Pre-printed using Scribe
Documentation	Labels	Sample identification	Pre-printed using Scribe
Documentation	Clear tape & Scissors	Label protection	
Sampling Tools	Bucket/Transfer Device	Sample transfer (if required)	Can also be used for sample homogenization
Sampling Tools	Sediment Scoop	Sediment sampling	Select based on analysis
Sampling Tools	Cooler	Sample containment	Cool to 4°C
Sampling Containers	Amber glass jars	Volatile Organics Analysis sample containment	Tight cap seal
Sampling Containers	50 ml HDPE (widemouth)	Metals analysis sample containment	
Reagents	10% Nitric Acid (HNO_3) solution	For decontamination of metals sampling equipment	Pre-mixed at lab
Reagents	10% Hydrochloric Acid (HCl) solution	For decontamination of organics sampling equipment	Pre-mixed at lab

Global Positioning System (GPS) - Trimble GeoXT 2008 Series

APPROVED:

07/16/13
ESAT Region 8 QA Coordinator — Date

7/16/13
ESAT Region 8 Team Manager — Date

7/16/13
EPA Task Order Project Officer — Date

7/16/13
ESAT Region 8 Task Lead — Date

DCN: EP8-7-7061

This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standard approach for United States Environmental Protection Agency (EPA) and Environmental Services Assistance Team (ESAT) Region 8 personnel to use a Global Positioning System (GPS) during field activities.

2.0 SCOPE AND APPLICATION

This SOP is specifically intended for application by EPA and ESAT personnel who conduct field work using a GPS.

3.0 SUMMARY OF METHOD

This SOP covers the use of TerraSync on Trimble GeoXT 2008 Series GPS machines as well as the Laser RangeFinder and Pathfinder Office. This SOP is based on manufacturers' instructions.

4.0 ACRONYMS AND DEFINITIONS

EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
GIS	Geographic Information System
GPS	Global Positioning System
HASP	Health and Safety Plan
SOP	Standard Operating Procedure

Geographic Information System (GIS): A system of hardware and software used for storage, retrieval, mapping, and analysis of geographic data.

Global Positioning System (GPS): A navigational system involving satellites and computers that can determine the latitude and longitude of a receiver on Earth by computing the time difference for signals from different satellites to reach the receiver.

Terrasync: Software used in GPS units that captures and stores spatial data collected in the field.

Trimble: A private GPS Company that provides equipment and technical support, including the 2008 GeoXT GPS units and TerraSync software.

5.0 HEALTH AND SAFETY

There are no health and safety issues requiring mention in this SOP; however, refer the applicable site-specific Health and Safety Plan (HASP) any time field work is conducted.

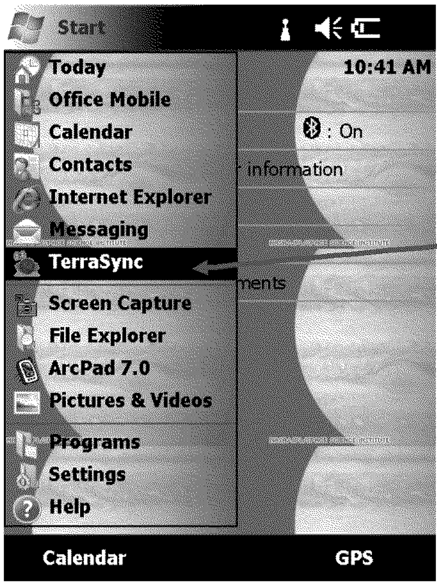
6.0 EQUIPMENT

Trimble GeoXT 2008 Series GPS unit with TerraSync
LTI TruPulse 360 Laser RangeFinder
TruPulse 200B/36B Cheat Notes card
Tape measure

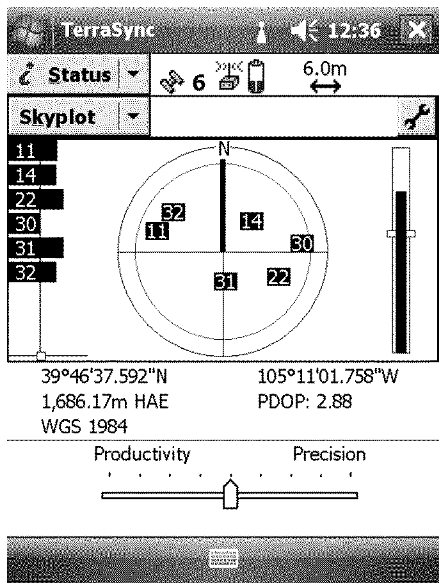
Flags
Computer and/or external hard drive

7.0 TERRASYNC

7.1 Starting TerraSync



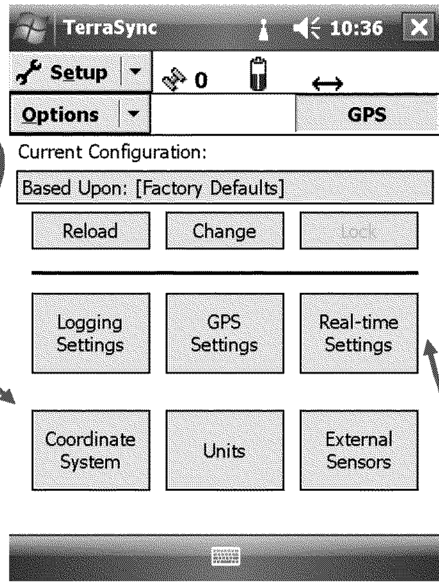
To begin, press the green button on the Trimble unit. Tap **Start**, then **TerraSync** to open the GPS application (left). If not available, tap **Programs**, then select **TerraSync**.

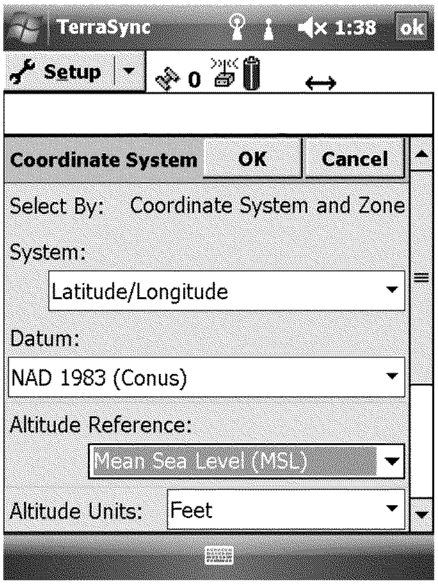


TerraSync will open with the **Status-Skyplot** page and begin to acquire satellite information. A minimum of four satellite locks are necessary to obtain a position. More satellites and proper satellite geometry increase accuracy.

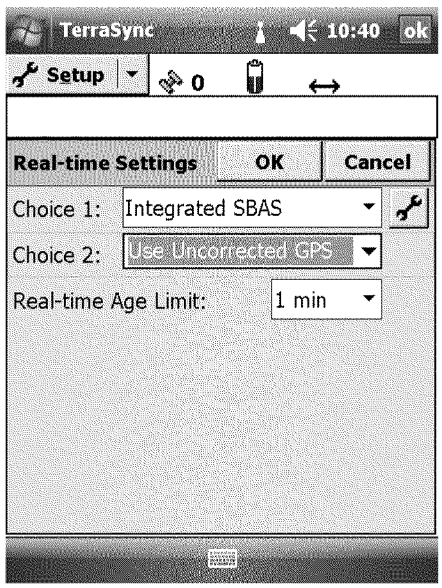
7.2 TerraSync Setup Menu

As TerraSync begins to acquire satellites, make sure the unit is properly configured. Tap the upper drop down menu and choose **Setup**. From the **Setup** page, tap **Coordinate System** and ensure that your screen matches the figure below. If a different coordinate system is required, use the dialog shown below to make necessary changes. Keep defaults if unsure. Select **OK** to return to setup menu.

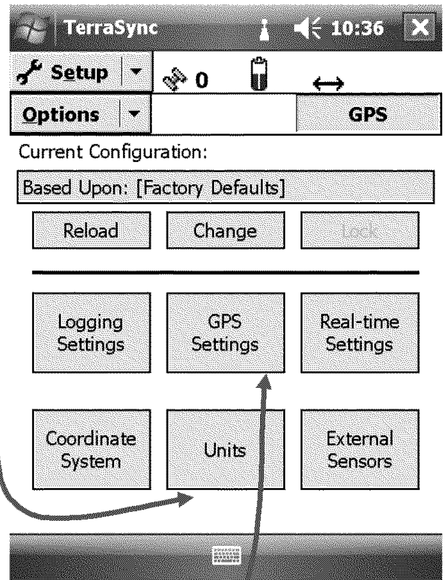


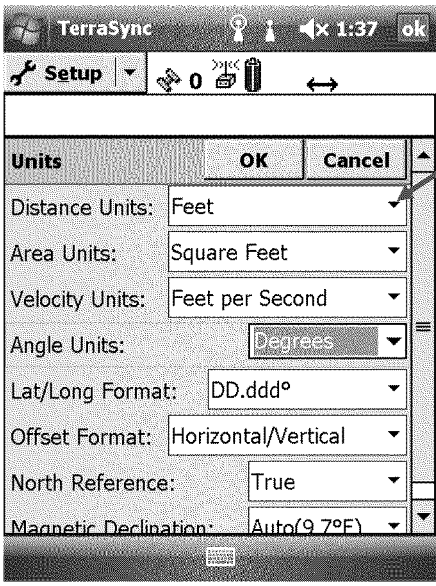


Next, choose **Real-Time Settings.** These settings will allow you to receive real time differentially corrected readings from satellite-based radio transmitters, if available. If not available, you will be using uncorrected GPS signals that can be corrected later by ESAT Region 8 GIS personnel. Please ensure that your screen matches the figure on the right. Select **OK** to return to setup menu.

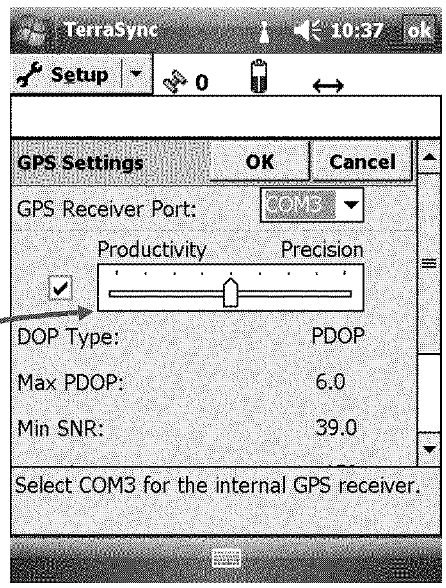


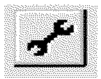
The **Units** menu allows you to choose which units will be used for display in the **Map** section (feet). Make any necessary changes. Select **OK** to return to setup menu.

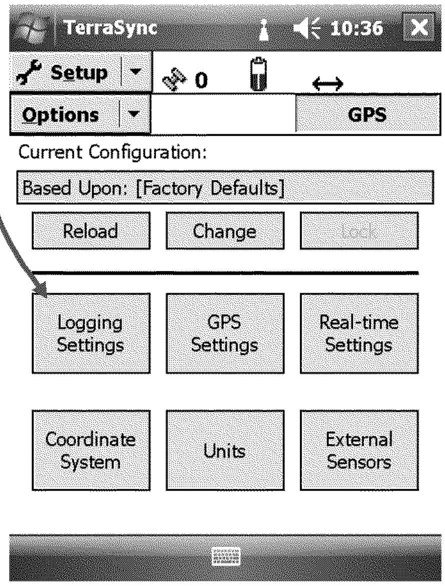


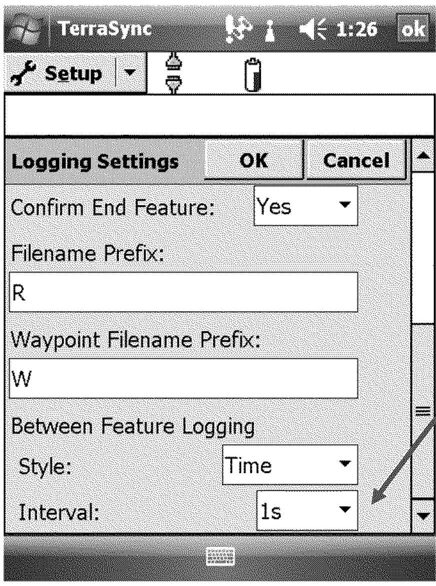


Under the **GPS Settings** menu, you can control the level of accuracy that your unit measures by adjusting the slider bar. Keep the defaults unless you are sure that you are willing to sacrifice precision for productivity or vice versa. Make sure that the **Productivity/Precision Bar** is set in the middle to give you a PDOP of 6.0, a minimum SNR of 39, and a minimum elevation of 15 degrees. Keep the settings for Velocity Filter and NMEA Output set to "Off." Select **OK** to return to setup menu.

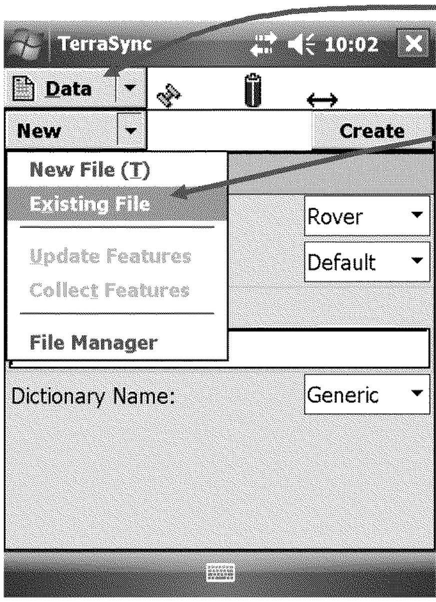


The **Logging Settings** menu allows you to adjust how you will log your data. In this dialog box, you will set your logging interval to either 1 or 5 seconds, depending on the data being collected (the default used by ESAT is 1 second; however, 5 seconds is better suited for high volume data collection, such as a large polygon or area). You will also set your antenna height to the height at which you will be collecting your data. Normally, this will be set at 1 meter (approximately 3.5 feet or the height an average person would hold the unit). If you wish to change Antenna Height, click the following icon located just right of the Antenna Height figure: 

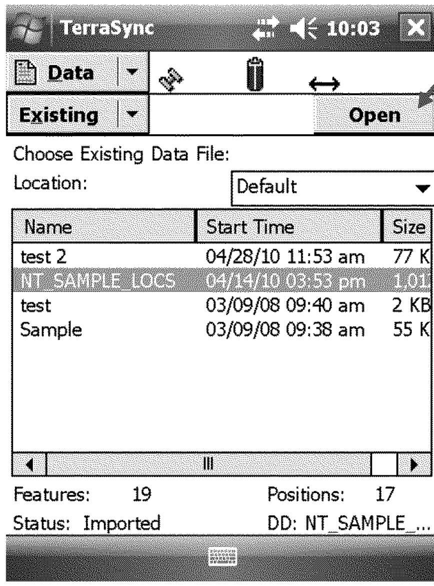




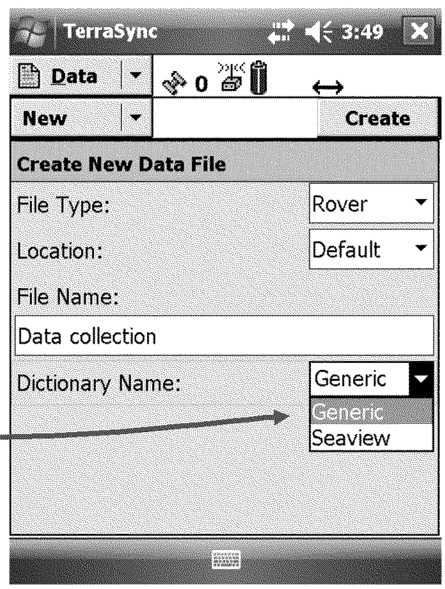
7.3 TerraSync Data Menu



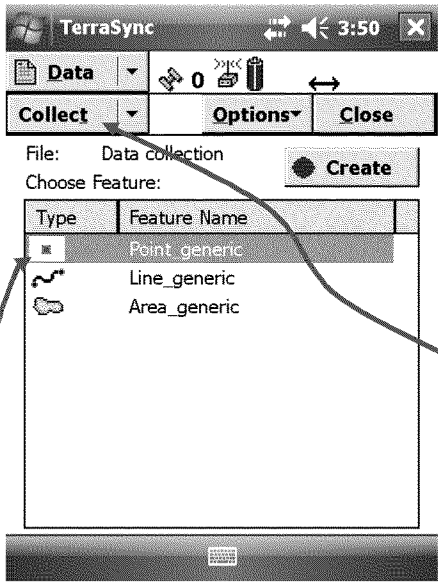
Select **Data** from the main drop down menu. The default will be to create a new data file. Click the drop down labeled **New** and choose **Existing File**. From this menu, select the appropriate file and click on the **Open** button. Then confirm antenna height.



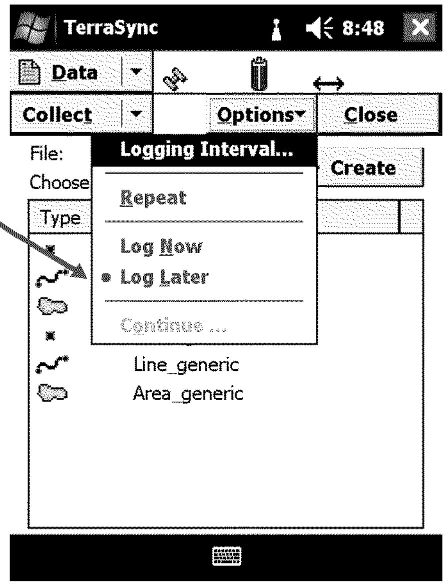
If you wish to create a new file or cannot find the appropriate existing file, click the **New** tab. Rename the new file to a project specific name. Select the proper data dictionary. Choose **Generic** if a data dictionary does not exist for your project. Tap **Create** and confirm antenna height at 1 meter (~3.5 feet) in subsequent pop-up.



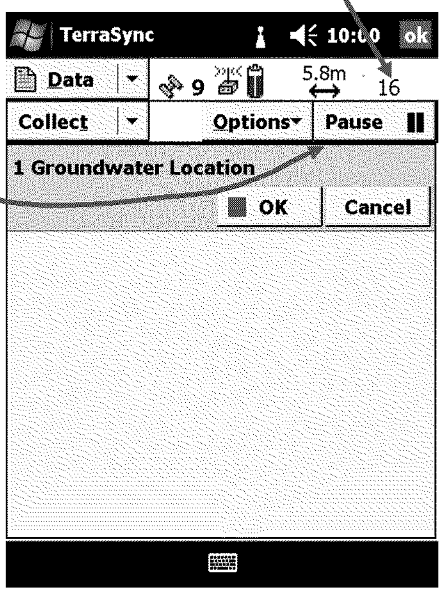
The features you are able to collect are now available in a list (below left). Click the **Options** tab and select **Log Later**. Use the **Options** tab to select a logging interval appropriate for the type of data you are collecting (below right).



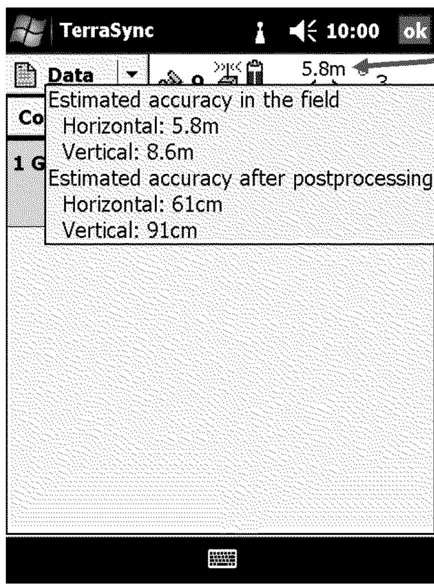
Ensure that this Tab has **Collect** selected.



Select **Point**, **Line**, or **Area,** then tap the **Create** button. When ready to collect data, tap the **Log** button (button will show pause if not currently logging, as shown here). Attribute any and all necessary fields (tap keyboard icon to enter description). This can be done before, during, or after collecting your data. Make sure to collect at least 30 positions for each point. Your positions count is displayed above the **Log** button.

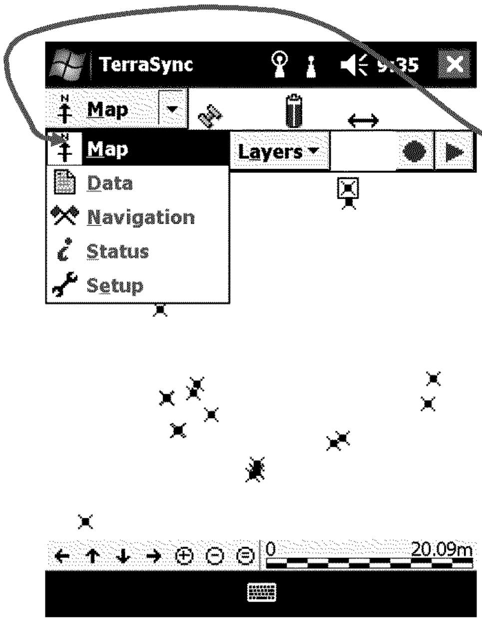


Estimated accuracy is listed here. By tapping this number more detail will be shown (right).

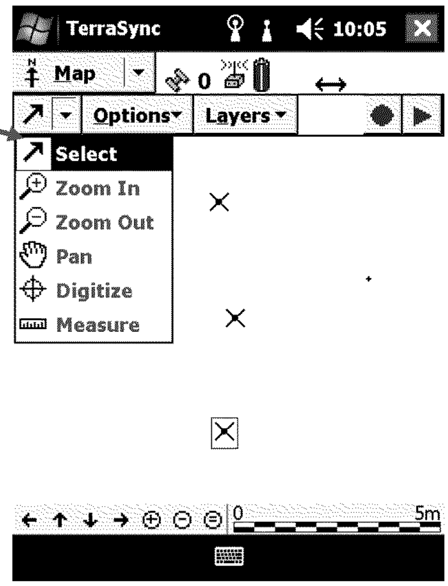


When ready, click **OK** to finish feature collection. You will be returned to the main data collection page. Continue collecting data until finished. Data will be saved as you go. When finished with data collection, exit out of TerraSync and turn off unit (Green button).

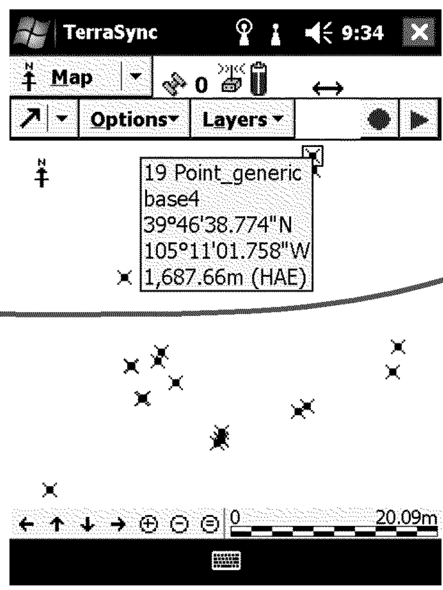
7.4 TerraSync Map Menu

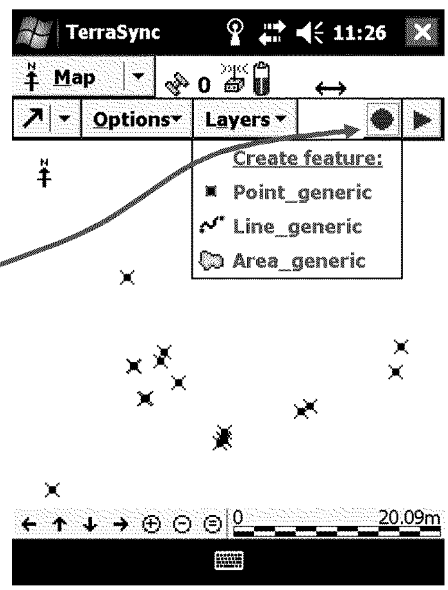


To view data that has been collected, choose the **Map** section. There are multiple tools to help with the display of data shown here.

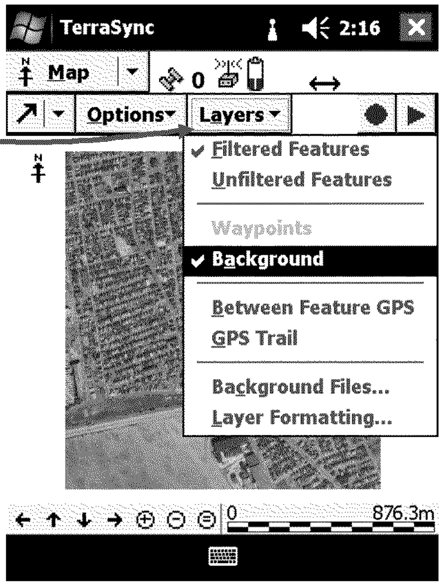


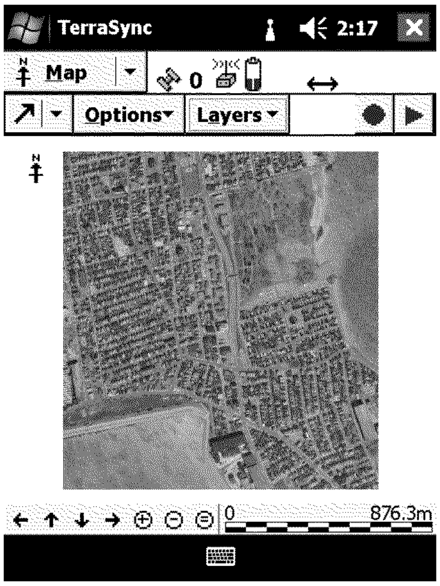
Tapping any point will display the attributes of a point, including a point's latitude and longitude. Logging GPS positions can be accomplished in the **Map** section. After selecting and setting up the proper data file in the **Data** section, choose **Map.** Tap **Create Feature** button, then tap the arrow key on the top right to begin logging points. When enough points have been collected, click **Ok**.





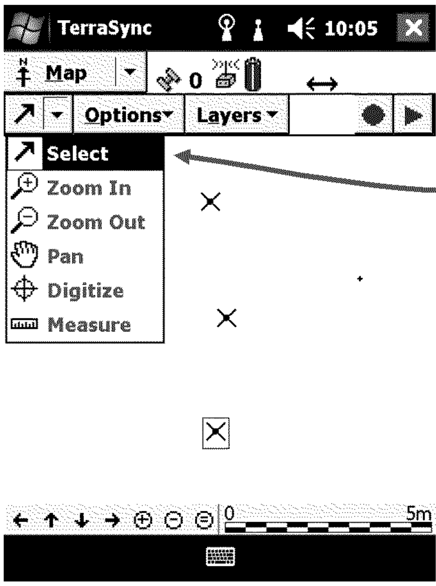
The on-screen display in the **Map** section can be enhanced with an aerial or topographical image. This must be set up prior to field collection with ESAT. If available select **Layers** and tap **Background** to enable the image (See Below).

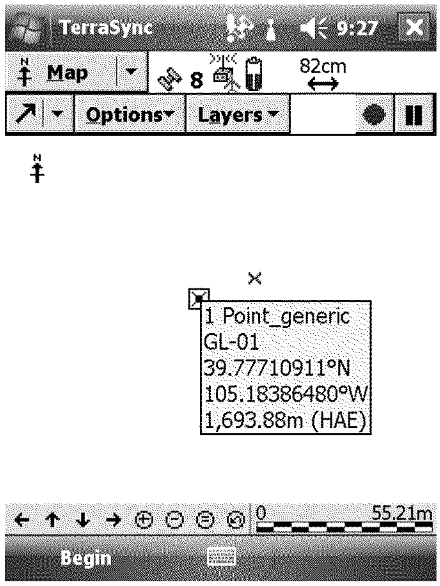




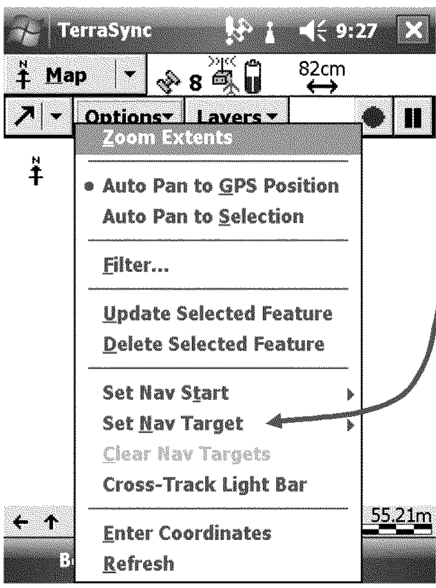
7.5 Navigation Using TerraSync

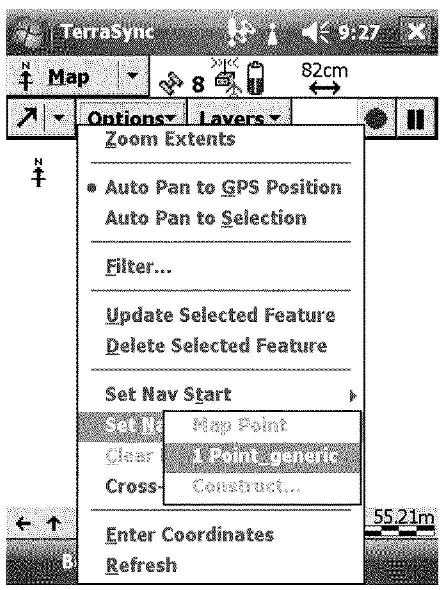
TerraSync and your Trimble unit can help you to navigate to a specific point if that point has already been loaded into the data. In the **Map** tab, use the selection arrow to select the point of interest (below, right).



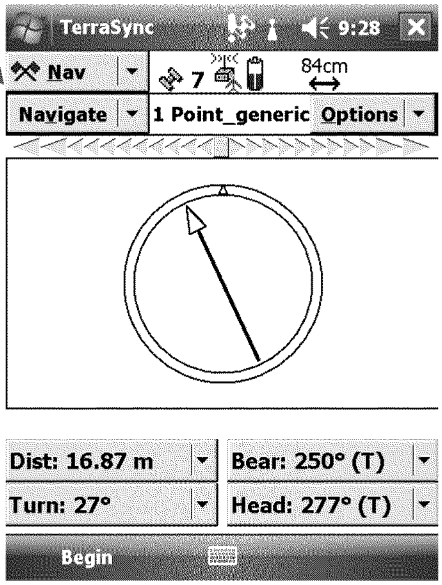


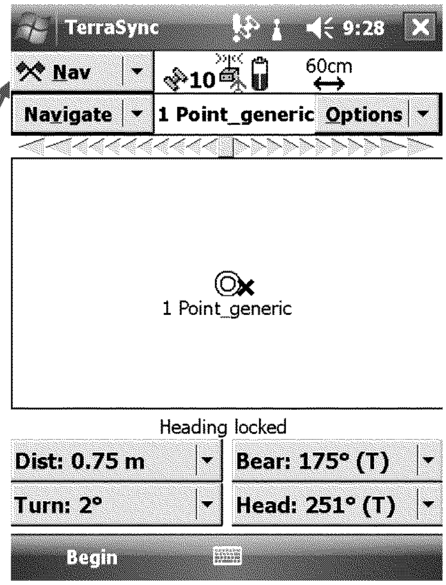
Tap the **Options** tab and then select **Set Nav Target.** Next, select the highlighted feature in the drop down menu (below). On the map, the point you are navigating to will have a crossed flag symbol like this:



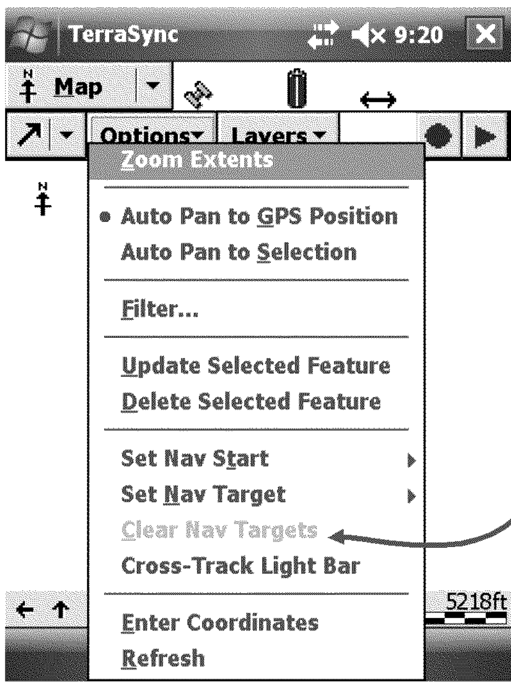


Once you have selected the point feature of interest, you can switch to the **Nav** menu on the main drop down list (left). The arrow guides you in the direction of the point of interest giving distance and bearing. You have to be moving for this to work. This navigation menu will change once you are close (right). You are the **X.**





Once finished navigating, go back to the **Map** section by going to the **Nav** menu and clicking **Map**. In the **Map** section, click **Options** and select **Clear Nav Targets.**



8.0 LASER RANGEFINDER

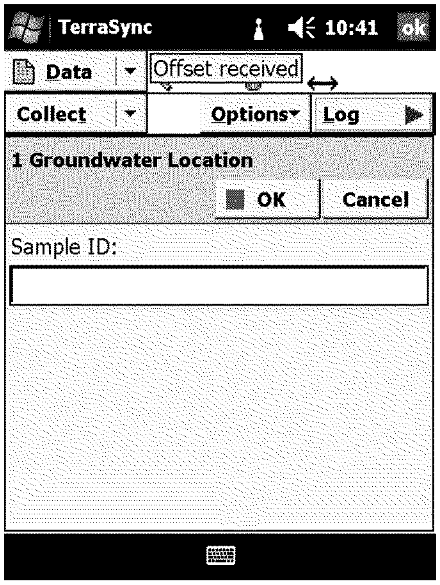
The LTI TruPulse 360 Laser RangeFinder is a measurement device that emits a laser to a distance of up to 1,000 meters and returns various information, such as vertical distance, horizontal distance, slope distance, inclination, azimuth, and height.

8.1 Enabling the Laser RangeFinder for Offset Point Collection

The Trimble units have been configured with the Laser setup and are ready to work. Laser #1 is setup for GPS units 1 & 2. Laser #2 is setup for GPS units 3 & 4. Before using the laser on your GPS device, you need to turn on Bluetooth on the Laser Rangefinder. To do this, look into the Laser eye piece and turn on the laser by clicking the **FIRE** button on top of the laser. Press the down arrow for four seconds. Press the down arrow once to get to the Blue Tooth option. Select **FIRE**. You have now enabled Blue Tooth on the laser. It should read **Bt_on**. Press **Fire** to return to main menu. Once the laser is enabled, it should be ready to work when you open a data file in the **Data** section of TerraSync.

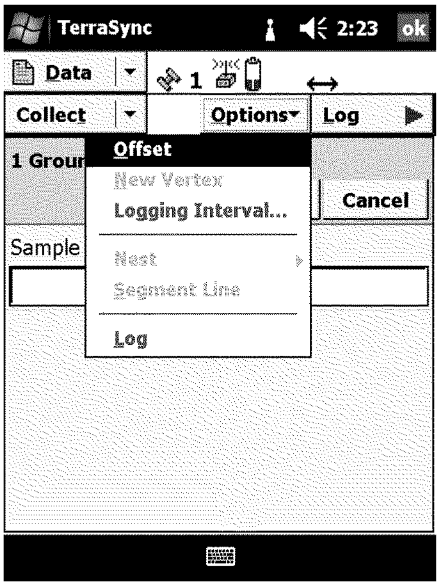
8.2 Using the Laser RangeFinder in TerraSync

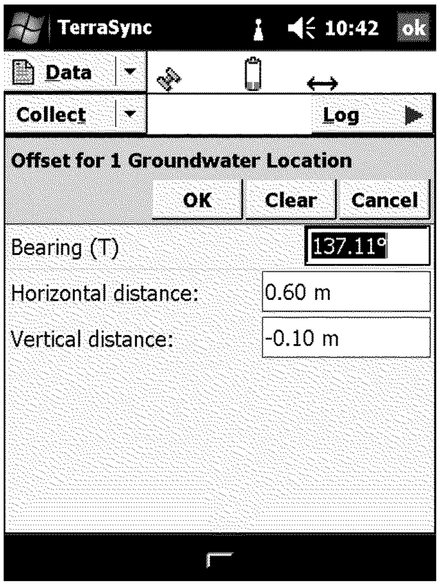
To use the laser once setup is complete, follow the same steps in data collection and begin logging points. After naming your sample ID, begin logging points. Aim the Laser RangeFinder at your target and push the **FIRE** button. You will be given notice that the offset has been received. Make sure that the **Offset received** notification appears. Sometimes your laser shot will not register and you will need to repeat your shot. When finished with the laser, it will turn off by itself.



REFER TO SECTION 8.3 TROUBLESHOOTING THE LASER RANGEFINDER IF YOU EXPERIENCE ANY DIFFICULTIES IN THE SETUP PROCESS.

To confirm the offset distance, you can tap the **Options** button and then click **Offset** to view the offset information that has been gathered.





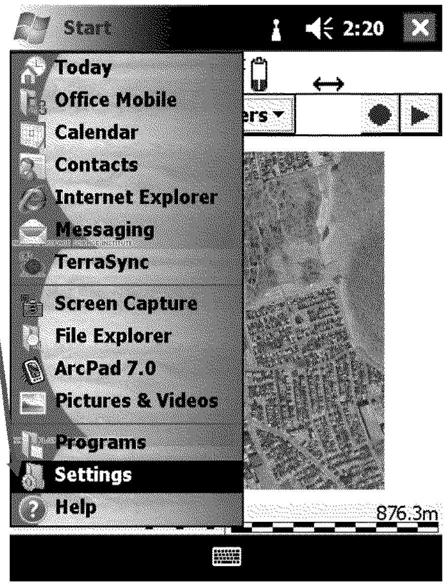
Your offset has been captured. Click **OK** and then click **OK** again to capture the offset point.

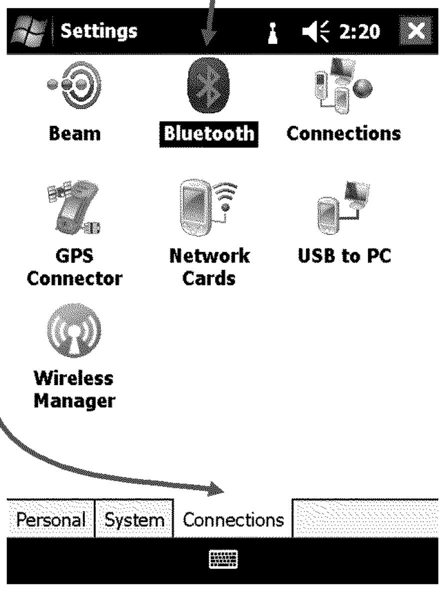
For additional setting and configuration options for the laser, refer to the TruPulse 200B/36B Cheat Notes card. This card can be found in the Laser Box.

8.3 Troubleshooting the Laser RangeFinder

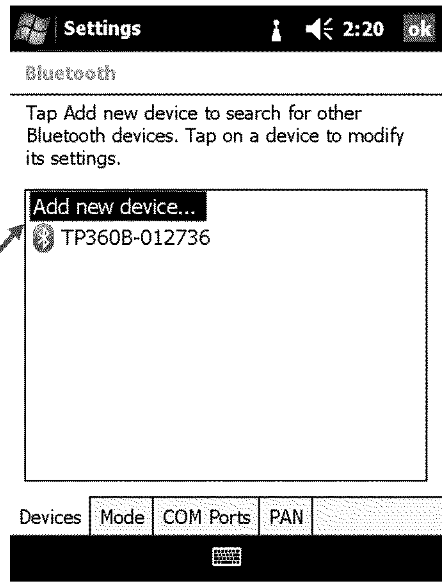
If enabling the laser did not work as described above, you will have to reestablish and reconfigure the Bluetooth connection to the Trimble unit. This is common so do not panic! At times, this process may seem to loop on itself. Follow these instructions and you will be ready to use the laser in very little time.

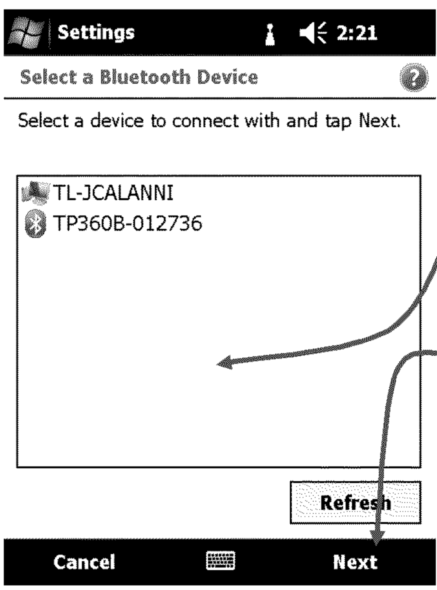
First ensure that the laser's Bluetooth is turned on.
To activate the laser on your GPS device, tap **Start** and select **Settings.** At the bottom of the **Settings** page, select the **Connections** tab, then tap the **Bluetooth** icon.



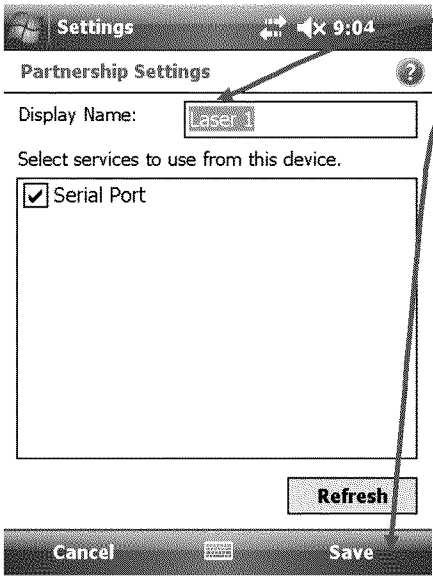


Choose **Add new device.**

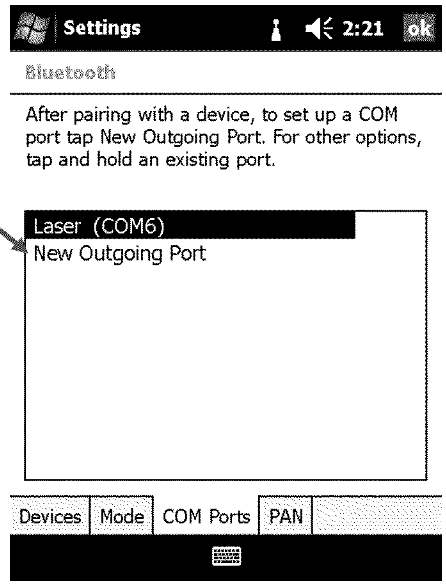




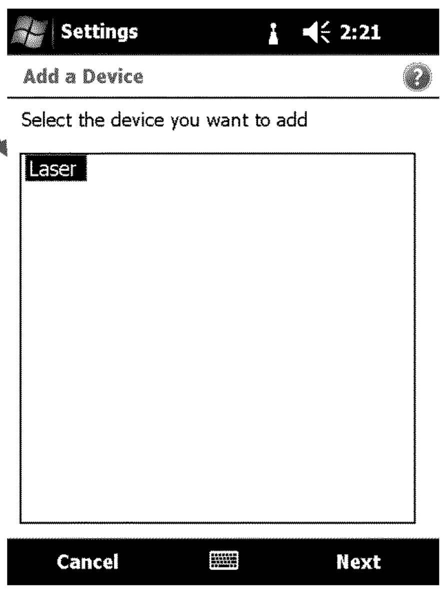
Select the Bluetooth Icon with label TP360B-012736. This number code is for Laser 1 (used with GPS unit 1 and 2); if using Laser 2 (for use with GPS units 3 and 4) the number code will be TP360B-017564. Tap **Next**. You will be prompted to enter the following pass code: 1111

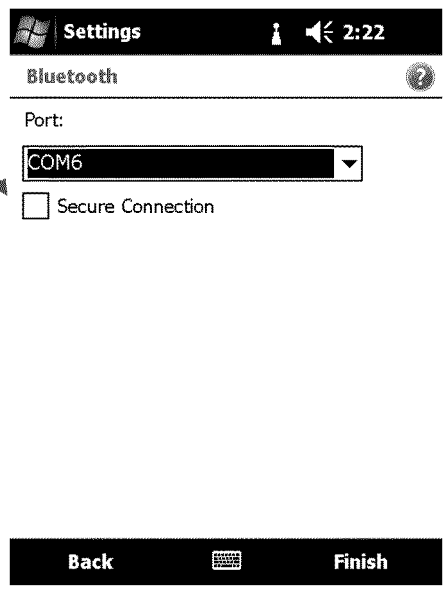


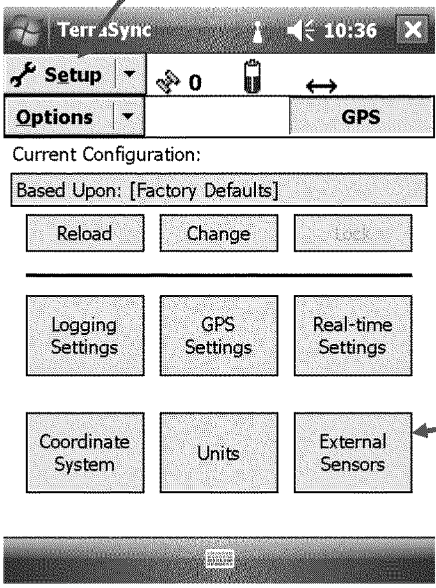
Check **Serial Port**, rename the **Display Name** "Laser," and click **Save** (left). In the next screen, choose **COM Ports** and select **New Outgoing Port**.



Select **Laser** and tap **Next**. On the next screen, choose a COM Port from the drop down. ***Do not check Secure Connection*.** Tap **Finish. Close this application**.

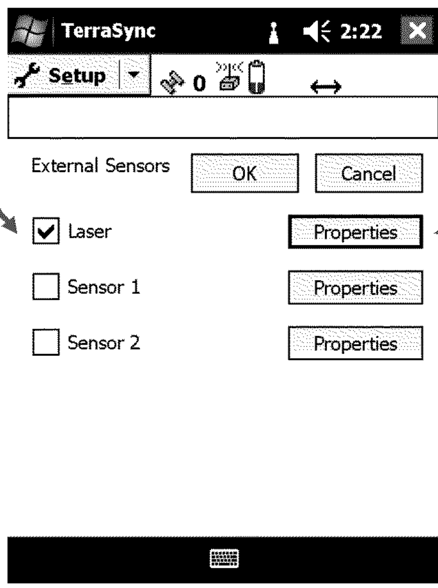




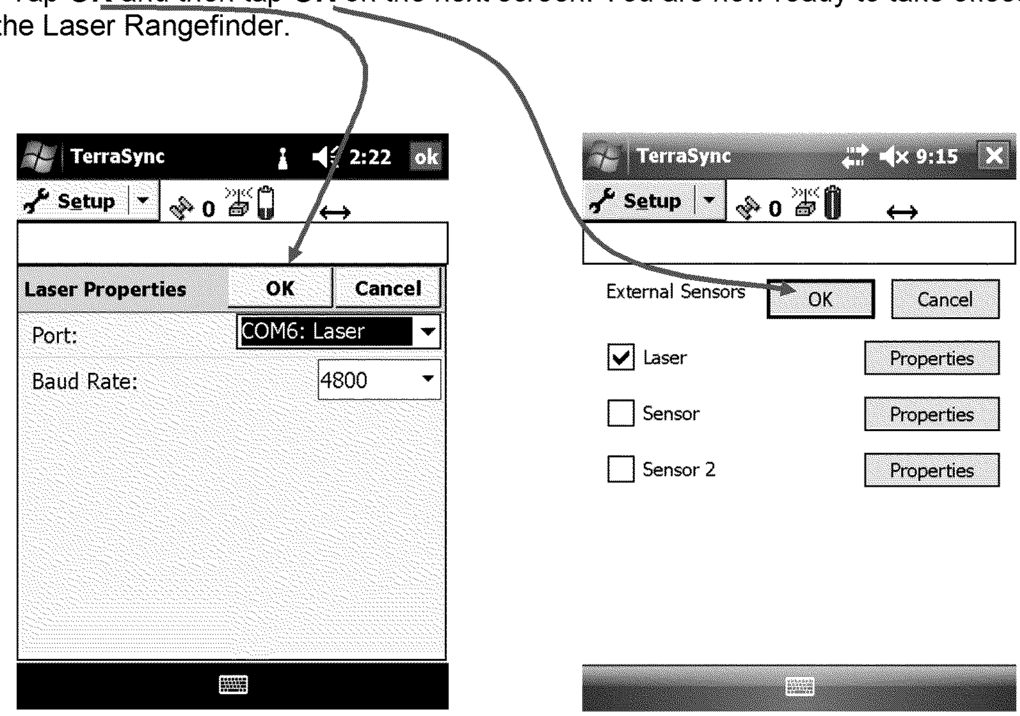


Back in TerraSync, tap the **Setup** menu and choose **External Sensors** to link to the Laser Rangefinder.

Check the box next to **Laser** to enable. Next tap the **Properties** box to setup the correct COM Port.



Tap the drop down box next to **Port** and choose the COM Port that you assigned to the **Laser**. Tap **OK** and then tap **OK** on the next screen. You are now ready to take offset points using the Laser Rangefinder.



9.0 TROUBLESHOOTING

If the unit does not show any satellites the GPS receiver may have been disconnected. Simply go into the **Setup** menu and click the **GPS** button to connect to the receiver. Sometimes the units go into deep hibernation and will not turn on using the on/off button. To fix this, the power button and tiny grey reset button need to be depressed simultaneously. Use a pen to press the reset button (inset on the upper left of the Trimble keypad).

9.1 Contact numbers for assistance

Trimble Support 1.800.728.5066

10.0 REFERENCES

Trimble Navigation Limited. Trimble Mapping and GIS Products: New Post-Processing Engine. 2009. Westminster, CO, USA.

Trimble Navigation Limited. TerraSync Software Reference Manual. 2008, Version 3.21, Revision A. Westminster, CO, USA.

Trimble Navigation Limited. GeoExplorer 2008 Series User Guide. 2008, Version 1.00, Revision B. Westminster, CO, USA.

Water Quality Measurements with the In-Situ® Multi-Parameter Meter

APPROVED:

ESAT Region 8 QA Coordinator — Date 06/06/12

ESAT Region 8 Team Manager — Date 6/6/12

ESAT Region 8 Task Lead — Date 7/10/12

EPA Task Order Project Officer — Date 6/8/12

DCN: EP8-7-7061

This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to document best practices in the use of the In-Situ® multi parameter water quality meter. For optimal performance, refer to the In-Situ® Multi-Parameter Troll® 9500 Operator's Manual and the Rugged-Reader® Operator's Manual.

2.0 SCOPE AND APPLICABILITY

This SOP is applicable only for In-Situ® water quality meters that are currently in use by United States Environmental Protection Agency (EPA) and Environmental Services Assistance Team (ESAT) field personnel. Water quality parameters may include pH, dissolved oxygen (DO), specific conductance, and temperature readings.

3.0 SUMMARY OF METHOD

Water quality measurements are a critical component of field sampling activities. Water quality meters are versatile tools for various measurements of water quality. For field sampling purposes, water quality meters are calibrated on site before use to account for barometric pressure. The main parameters for surface water quality are pH, DO, specific conductance, and temperature. These parameters are built in to the In-Situ® Rugged Reader® Troll® device and it's Pocket Situ 4 Software. Once calibrated, sampling teams have up to 24-hours to record water quality readings if barometric pressure conditions remain constant (see section 11.2). Data is recorded in a site-dedicated field logbook.

4.0 ACRONYMS AND DEFINITIONS

mg/L	milligram/liter
mL	milliliter
µS/cm	microsiemens/centimeter
DO	Dissolved Oxygen
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response Standard
OSHA	Occupational Safety and Health Administration
PPE	Personal Protective Equipment
SAP/QAPP	Sampling and Analysis Plan/Quality Assurance Project Plan
SOP	Standard Operating Procedure

<u>Health and Safety Plan (HASP)</u>: A site-specific plan that outlines safety hazards and hazard mitigation practices.

<u>Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPP)</u>: Site-specific documents that outline sampling and data quality objectives for a field sampling project.

<u>Standard Operating Procedure (SOP)</u>: A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

5.0 HEALTH AND SAFETY

When working with potentially hazardous materials or in hazardous situations, personnel must understand and comply with the site-specific Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPP) and Health and Safety Plan (HASP) before the sampling event begins. When taking water quality readings in streams or surface impoundments containing known or suspected hazardous substances, adequate personal protective equipment (PPE) such as nitrile gloves, safety glasses, and waders, are necessary to prevent exposure. If entering a stream, safety equipment, including a personal floatation device and non-slip footwear should be worn in addition to general PPE. When taking measurements from a vessel in an impoundment or flowing waters, appropriate safety procedures and practices should be followed.

6.0 CAUTIONS

To ensure safe and reliable operation of the equipment, the manufacturer's directions for transport, cleaning, decontamination, storage, and operation should be followed. See the operations manuals for additional guidance (Multi-parameter Troll® 9550 and the Rugged Reader®)

7.0 INTERFERENCES

Improper calibration of the instrument can lead to erroneous readings or equipment malfunction. For each calibration standard, be sure to condition the instrument/probe with a rinse of the appropriate standard before calibration. Additionally, the hand held Rugged-Reader® unit should be fully charged before deployment to the field. If the unit fails to recognize the probe, make sure to check the batteries located in the upper part of the probe just below the interface cable connection. Note that batteries do not operate optimally in cold weather conditions.

8.0 PERSONNEL QUALIFICATIONS

Any personnel involved with field sampling activities must be cleared for participation by their organization's health and safety officer. Clearance includes medical monitoring, respirator fit testing, and Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response Standard (HAZWOPER) 40-hour training. Personnel who will be collecting surface water quality data must be familiar with this and any other relevant SOPs, including the Sample Equipment Decontamination SOP FLD 02.00, Sample Preservation SOP FLD 03.00, Surface Water Sampling SOP FLD 01.00, Sample Custody and Labeling SOP FLD 11.00, and General Field Sampling Protocols SOP FLD 12.00.

9.0 EQUIPMENT AND SUPPLIES

Equipment needed for collection of surface water quality data may include:

Water quality collection device – Multi-parameter Troll® 9550 and the Rugged-Reader®

PPE - personal floatation device, waders, gloves, proper footwear, safety glasses, insulating clothing for cold water, etc.

Mapping and location tools – Global Positioning System (GPS) units, site/local area maps, compass, tape measure, survey stakes, pin flags, camera, 2-way radios

Documentation – field logbook or field data sheet(s)

Calibration standards - see Section 10.0, Standards and Reagents

10.0 STANDARDS AND REAGENTS

Certified standards are required for calibration of the Rugged Reader® In-Situ® water quality multi-parameter meter. pH calibration can be done at one to three points using buffer solutions at pH 4, 7, or 10. Specific conductance is calibrated with one point, usually at 1000 µS/cm. In addition to certified standards, it is best practice to also have spent standard, referred to as rinse, to use as probe conditioner in order to equilibrate the sensor. Refer to the Sampling Equipment Decontamination SOP FLD 02.00.

11.0 PROCEDURES

Commonly used procedures for the use of the Rugged Reader® In-Situ® water quality meter are provided in the following sections. Note that this equipment has many other functions, but only the most frequently used procedures for EPA Region 8 field activities will be discussed. For further information on potential uses and capabilities, consult the Rugged-Reader® and Troll® 9550 Operations Manuals.

11.1 Pre-Deployment Activities

Before field deployment, it is important to check the equipment for functionality. The batteries to the Rugged Reader® should be charged at least 24 hours prior to use. The Troll® 9500 requires two D-cell batteries. Calibration frequency varies between sensor types on the multi-parameter meter and environmental conditions. Consult calibration frequency requirements mandated by sampling event protocols or project data quality objectives. It is recommended that water quality sensors be stored, calibrated, and maintained in accordance with the In-Situ® multi-parameter Troll® 9500 operations manual. Sufficient time for thermal stabilization of standards to ambient conditions should be considered before calibration. To reduce the time for stabilization, all calibration standards and the In-Situ® equipment should be stored at the same temperature before starting calibration.

11.2 Field Water Quality Measurements – Calibration

Many sampling sites in EPA Region 8 are located at various elevations with differing barometric pressures, and as a result, re-calibration of the water quality sensors will occur at a greater frequency than manufacturer recommendations. Upon arrival on a site, it is recommended that a 500mL bottle of tap water be used to begin aeration of the optical DO sensor. Specific conductance and pH calibration can occur while the water aerates. In general, most mine effluent sites have pH values between 4.0 and 7.0; therefore, only a 2-point calibration with 4.0 and 7.0 pH buffer solutions is necessary. If deployed to a site where pH values are completely unknown, perform a 3-point calibration (adding the 10.0 calibration). Below are calibration procedures for pH, specific conductance, and DO measurements.

11.2.1 pH Calibration

Connect the Troll® 9550 to the Rugged-Reader® with the interface cable and power on the unit. From the starting screen, tap **Start** (located on the upper right corner of the screen) with the stylus. The Start menu is the main access point to all programs, files, and settings loaded on the Rugged-Reader®. If this is the first time the Troll® 9550 is connected to the Rugged-Reader®, follow the instructions for using the Connection Wizard in the Rugged-Reader® Operations Manual (for subsequent uses, tap on the COM1-19200 line in the navigation window). If the Troll® 9550 is not recognized in the navigation window you may have to 'find' the device. Do this by tapping the COM port (labeled as COM1-19200) and then tap **Find** located in the lower right hand corner. The information for the Troll® 9550 should appear in the Rugged-Reader® navigation screen. If there is no connection to the Troll® 9550, check cable connections and/or battery status. Once the Troll® 9550 connection appears as a line in the navigation window, tap to open the **Parameters** option. Tap this twice in order to bring up the sensors that are currently connected to the probe (this should always be pH, specific conductance, DO, temperature, and barometric pressure). From this menu, tap **pH**. Three options will appear at the bottom of the screen, select **Calibrate**.

1. This will bring up the calibration setup menu. Select number of calibration points desired (usually 2). Select **Next**.
2. Start the pH calibration with the lowest standard. Use the pH 4.0 standard rinse to condition the sensor, empty the cup, and then place certified pH 4.0 standard in the calibration cup up to the fill level line. Press **Run**.
3. The sensors will take a few moments to stabilize for temperature and pH. Once stabilized, the screen will immediately prompt for the next standard. Follow the same procedure by first conditioning the sensor with the pH standard rinse, then fill the calibration cup with the certified standard. Press **Run** again.
4. Once the final calibration has stabilized, the user will be given the option to view the calibration report. Click **Yes** to view the report and record the final calibration results in the instrument-dedicated calibration logbook.

11.2.2 Specific Conductance Calibration

To calibrate for specific conductance, select the **Conductivity** option from the parameters menu by tapping it once, then select **Calibrate** by tapping it once. This will bring up the calibration setup menu.

1. This menu allows the user to select the standard intended for use. If the standard being used does not appear as one of the options, select **Other** to input the desired standard. Tap **Next**.
2. Condition the sensors by saturating with specific conductance rinse of similar concentration, and then fill the calibration cup with the certified standard. Make sure to fill the cup all the way to the fill line.
3. Once the calibration is stabilized, the user is given the option to view the calibration reports. Select **Yes** and record the new calibration results in the instrument-dedicated calibration logbook.

11.2.3 Dissolved Oxygen Calibration

Finally, the optical DO sensor should be calibrated. If the other parameters were calibrated first, there should have been sufficient time to saturate with oxygen (10-15 minutes). From the parameters menu, tap once on the **Rugged Dissolved Oxygen** icon, then once again on the calibration option at the bottom of the screen.

1. The first screen allows the user to edit the barometric setting. Select **Yes**.
2. The next screen will give the user options as to how the barometric setting should be calculated into the DO calibration. Select the **Use Vented Cable** option, and then tap **OK**.
3. The following screen gives the user the choice of 1) restoring default settings and calibrating, 2) restoring defaults and not calibrating, and 3) calibration only. Select the **Calibrate Only** option, and then tap **Next**.
4. Place the DO probe in the saturated tap water, then tap the **Run** button. The sensor will take a few moments to equilibrate for temperature and DO.
5. Once stabilized, the unit will prompt for a zero oxygen calibration. To skip this, simply tap the **Next** button. To finish the calibration, tap the **Finish** button.
6. The user will be given the option to see the calibration reports. Select **Yes** and record the calibration information in the instrument dedicated logbook.

11.3 Field Water Quality Measurements – Data Collection

Once calibration is complete, the user will be returned to the parameters screen. To begin collecting water quality data, tap the **Parameters** icon once, then the **Profiler** option once. The next screen will ask the user to assign a test name. Enable the keyboard icon on the bottom of the screen and type in the Location ID for the site that is to be sampled. Once finished entering the Test Name, tap **OK**. The screen will then display temperature, pH, specific conductance, DO, and barometric pressure data. Tap the icon on the bottom right of the screen that says **Continuous**. Allow several minutes for the readings to stabilize. Note: make sure the readings are within a certain range of expectation. Refer to previous readings at the site in the logbook to verify correct data ranges. Once the readings have stabilized, tap the icon **Stop Log**. Before exiting out of the screen, record the readings in a project dedicated field logbook. Data should be recorded in the following units for each parameter: pH, standard units; DO, mg/L; specific conductance, µS/cm; and temperature, °C. Once the data has been recorded in the logbook, tap the icon **Close** on the bottom right of the screen. To begin collecting data at a different site, repeat these steps found here in section 11.3.

11.4 Troubleshooting

When problems are noted during the calibration procedure, check the following:

1. Make sure the parameter shown on the screen is the same parameter to which the unit is being calibrated.
2. Make sure the proper standard is being used for its corresponding calibration value (i.e. make sure not to calibrate specific conductance with a pH standard).
3. Verify that the standards used have not been contaminated.
4. Make sure the equipment and solutions are at the correct temperature for the

calibration being performed.

5. Be sure to fill the calibration cup according to directions in the Troll® 9550 operations manual.
6. Make sure the pH reference electrode was filled with the proper pH filling solution.
7. Check the multi-probe housing and sensors for physical damage (cracked or bent electrodes) and fouling (tarnished, soiled, color change, or otherwise coated electrodes).
8. Check the multi-probe battery status. Recharge the unit if there are only one or two bars showing in the battery icon in the opening screen. There is also a battery voltage indicator under the parameters screen in the Pocket-Situ 4 software. If the Troll® 9550 batteries are running low, quit the Pocket Situ 4 software and replace the D-cell batteries.
9. If the above troubleshooting tips and maintenance procedures do not solve the problem, contact In-situ® technical support at 1-970-498-1500 or toll free at 1-800-466-1500 (Option 3).
10. If problems persist, refer to the In-situ® operator's manual. All In-situ® cases will include a copy of the operator's menu.

12.0 DATA RECORDS AND MANAGEMENT

All data and measurements must be recorded in the site-dedicated field logbooks. This data will be entered into a spreadsheet and published in Scribe. Any recording errors should be struck out with a single line through the incorrect value, initialed by the recorder, and then re-recorded with the correct value. All calibration or repair data should be recorded in the equipment-dedicated field logbook.

13.0 QUALITY CONTROL AND ASSURANCE

EPA and ESAT Region 8 personnel who use these instruments will calibrate the In-situ® each day before use and will perform maintenance procedures as needed. If personnel feel uncertain about the quality of measurement data in the field, performance of the instrument will be checked using a known calibration standard. Corrective actions will be determined on a case by case basis with all circumstances being considered. Data will be flagged as needed in the final data package.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

In-Situ, Inc. Multi-Parameter TROLL® 9500 Operator's Manual. Rev. 007, January 2009.

In-Situ, Inc. Rugged Reader® Operator's Manual. October 2007.

Pore Water Sampling

APPROVED:

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ESAT Region 8 Team Manager — Date: 6/6/12

EPA Task Order Project Officer — Date: 7/10/12

ESAT Region 8 Task Lead — Date: 6/8/12

DCN: EP8-7-7061

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures, methods, and considerations to be used when obtaining a pore water sample using PushPoint® samplers.

2.0 SCOPE AND APPLICABILITY

This document describes procedures for pore water sampling using PushPoint® samplers and is based on the Operators Manual and Applications Guide provided by MHE Products (Ver. 2.01,2/15). It is intended to be used by field personnel when collecting and handling samples in the field. All deviations from this SOP must be noted in the site-dedicated field logbook.

3.0 SUMMARY OF METHOD

Sediment pore water is collected using a pore water extractor, called a PushPoint® (Figure 1) which is made out of stainless steel tubing developed by MHE Products. The sampling end of the PushPoint® is inserted into the sediment to the desired depth, and pore water is extracted using a syringe or peristaltic pump.

4.0 ACRONYMS AND DEFINITIONS

COC	Chain of Custody
DOC	Dissolved Organic Compounds
DOT	United States Department of Transportation
DQO	Data Quality Objective
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
IATA	International Air Transportation Association
OSHA	Occupational Safety and Health Administration
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SVOC	Semi-Volatile Organic Compound
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

Health and Safety Plan (HASP): A site-specific document outlining potential safety hazards and hazard mitigation techniques.

Occupational Safety and Health Administration (OSHA): An agency that regulates health and safety standards in the United States.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

5.0 HEALTH AND SAFETY

Proper safety precautions must be observed when collecting pore water samples. Refer to Environmental Services Assistance Team (ESAT) site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When following this SOP, minimize exposure to potential health hazards in the field by using personal protective equipment (protective clothing, eye wear and gloves). Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

6.0 SAMPLE MANAGEMENT

The following precautions should be considered when collecting pore-water samples:

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.

- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party (see SOP FLD-11.00, or current version, “Sample Custody and Labeling”).

- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.

- Shipped samples must conform to all United States Department of Transportation (DOT) and/or International Air Transportation Association (IATA) hazardous materials shipping requirements.

- Documentation of field sampling is done in a bound logbook.

- Chain of custody (COC) documents must be filled out and remain with the samples until custody is relinquished until analysis is complete (or samples are disposed).

- All shipping documents, such as bills of lading, etc., shall be retained by the project leader and stored in a secure place.

7.0 INTERFERENCES

The following sections describe potential interferences when sampling for trace level contaminants. For decontamination procedures, see the Sampling Equipment Decontamination SOP FLD 02.00.

7.1 Potential Volatile Organic Analysis (VOA) Sampling Interferences

Pore water samples for volatile organic compound (VOC) and semi-volatile organic compounds (SVOC) analysis must be collected in 40-ml amber glass vials with Teflon® septa. The vials may be preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two week holding time, whereas, unpreserved samples have only a seven day holding time. Normally, either preserved or unpreserved vials can be used, but there are instances where the use of unpreserved vials is preferred. For example, if the surface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

Samples for VOC and SVOC analysis must be collected using either stainless steel or Teflon® equipment. Samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial, and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be refilled. Care should be taken not to flush any preservative out of the vial during topping off. If bubbles are still present after attempting to refill and cap the vial, a new vial should be obtained and the sample re-collected.

7.2 Potential Dissolved Metals or Dissolved Organic Compound Sampling Interferences

If a dissolved metals or Dissolved Organic Compounds (DOC) pore water sample is to be collected, in-line filtration or post-collection filtrations are acceptable approaches. The in-line filter apparatus uses disposable, high capacity filter cartridges (barrel-type) or membrane to filter the sample. The high capacity, barrel-type filter works well due to the higher surface area associated with this configuration. Post-collection filtration involves two approaches. The first approach is to take the sample water and filter it through a .45 micron filter apparatus. The second approach involves the use of a syringe with a .45 micron acrodisc filter attached to end of syringe.

Potential differences could result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations, including filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloidally-associated trace elements, and concentration of organic matter. Therefore, consistency of sample technique and filter characteristics is critical in the comparison of short-term and long-term results.

7.3 Special Precautions for Trace Contaminant Pore Water Sampling

1. A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled, and the gloves should be donned prior to handling sampling equipment and sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.

2. Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately from samples suspected of only having trace levels of contaminants.

3. All background or control samples shall be collected and placed in separate ice chests or shipping containers. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.

4. Samplers must use new, verified, certified clean disposable equipment, or pre-cleaned non-disposable equipment.

8.0 PERSONNEL QUALIFICATIONS

Any personnel involved with field sampling activities must be cleared for health and safety. Clearance includes medical monitoring, respirator fit testing, and Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. Personnel who will be collecting pore water samples must be familiar with this SOP and any other relevant SOPs, including the Sample Equipment Decontamination SOP FLD 02.00, Sample Preservation SOP FLD 03.00, Water Quality Measurements with the In-Situ® Multi-Parameter Meter SOP FLD 09.00, Sample and Labeling SOP FLD 11.00, and General Field Sampling Protocols SOP FLD 12.00.

9.0 EQUIPMENT AND SUPPLIES

The PushPoint® sampler consists of a tubular 3/16 stainless steel body with a screened zone at one end and a sampling port at the other. It comes with a guard rod that is nested in the tube during deployment in order to provide structural support and to prevent plugging and deformation of the screened zone (Figure 1). The screened zone consists of a series of interlaced machined slots that form a short screened zone with approximately 20% open area. Additional filters can be placed over the screened zone if additional screening is needed. Pore water is collected through the opposite end of the PushPoint® sampler through peristaltic flexible tubing using a syringe through the sampling port. Tygon® tubing is the preferred tubing to be used with PushPoint® samplers. However, other tubing can be used, if allowed by data quality objectives (DQOs) for the specific application. PushPoint® samplers can be custom made to any width or length.

There are many modifications that can be incorporated into the procedure to satisfy DQOs for a specific application. The procedures discussed in the following sections provide guidance on the basic operation of the PushPoint® and issues to consider when deploying the PushPoint® sampler to collect pore water.

Other equipment used in the process of pore water sample collection includes syringes, flanges, tubing, sample bottles or containers, and filters.

10.0 STANDARDS AND REAGENTS

Reagents will be used for preserving samples and for decontaminating sampling equipment (refer to the Sampling Equipment Decontamination SOP FLD 02.00 and Sample Preservation SOP FLD 03.00). The preservatives required are determined by the analysis to be performed and will be specified in the Sampling and Analysis Plan (SAP)/Quality Assurance Project Plan (QAPP), but usually include nitric acid (total recoverable and dissolved metals samples), hydrochloric acid (VOC samples), and phosphoric acid (DOC samples). The sampler should also be aware of any special sampling considerations, contamination issues, and sample compositing and mixing methods that could affect their sampling efforts. Appropriate regional guidance and procedures should be consulted for detailed sample collection, preservation, handling and storing, equipment decontamination, and quality assurance/quality control (QA/QC) procedures. The sampler should preserve and immediately cool all water samples to 4°C (±2°C) upon collection and samples should remain cooled until the time of analysis (do not freeze water samples).

11.0 PROCEDURES

It is critical in the collection of pore water to avoid surface water intrusion. Water will flow in a path of least resistance. If space is created around the sides of the PushPoint® sampler during deployment, surface water may flow down the outside of the tube to the screened area and into the intended sample. Therefore, the PushPoint® can be used with a sampling platform or flange (Figure 2), especially when collecting pore water near the sediment-surface water interface. However, if pore water is collected from deep in the sediments or in cobble-bottom streams, a flange may not be necessary. Additionally, it is important to note that a platform is only useful in specific situations when you are sampling multiple holes and specific depths and when sampling at shallow depths where the integrity of the hole may be a concern. It is critically important to collect samples from the hyporheic zone, or the area beneath the streambed where shallow groundwater is mixed with surface water (this area is critical to benthic macro-invertebrates and fish spawning activity). When inserted though the sampling platform, or flange, the flange should fit securely around the PushPoint® to eliminate surface water intrusion from around the PushPoint® body during sample collection.

The flange can be made of any material that will not cross contaminate the intended sample. If full scan analytical analysis is required, the flange should be made of inert material such as stainless steel or Teflon®. The size of the flange depends on the volume of pore water to be collected. If large volumes of pore water are to be collected, use a large flange size. If it is not practical to use a large flange, then multiple PushPoints® with smaller flanges can be deployed and smaller volumes can be collected from several PushPoints® for a composite sample. If multiple PushPoints® are deployed, they should be spaced at least 30 cm apart.

11.1 PushPoint® Sampler Basic Operation

The PushPoint® sampler should be inserted into the sediment as carefully as possible (Figure 2). When deploying the PushPoint®, care must be taken not to disturb the sampling area. If the sampler is wading in the water body, the sampler should lean out and insert the PushPoint® as far as possible away from where he/she is standing in order to reduce potential effects of the sampler on the integrity of the pore water sample. Depth of penetration of the PushPoint® into the sediment depends on the objectives of the specific investigation. Once depth is established for sample collection, be sure to measure and record the sampling depth in the logbook.

After the PushPoint® has been deployed, carefully remove the guard rod and attach the sample tubing (Figure 3). The other end of the sample tubing can be connected to the sample withdrawing device, such as a peristaltic pump or syringe (Figure 4). Before collecting a pore water sample, be sure to purge out all air and surface water from the PushPoint® sampler and sample tubing with the appropriate amount of pore water. At least three volumes of pore water (until water is clear) should be purged before sample collection.

11.2 Peristaltic Pump/Vacuum Jug Collection

The peristaltic pump/vacuum jug can be used for sample collection because it allows for sample collection without the sample coming in contact with the pump head tubing. This is accomplished by placing a Teflon® transfer cap assembly onto the neck of a clean standard 1-liter amber glass container. Teflon® tubing (3-inch outside diameter) connects the container to both the pump and the sample source. The pump creates a vacuum in the container, thereby drawing the sample into the container without it coming into contact with the pump head tubing.

Because the sample is exposed to a vacuum and is agitated as it enters the vacuum jug, this method cannot be used for collection of VOC samples. An alternative method for collecting VOC samples involves filling the Teflon® tubing with sample by running the pump for a short period of time. Once the tubing is full of water, the tubing is removed from the PushPoint® and, after the tubing is disconnected from the pump head tubing, the water is allowed to drain, by gravity, into the sample vials. Alternatively, without disconnecting the tubing from the pump head, the contained sample can be pushed out of the tubing and into the sample vials by reversing the peristaltic pump at low speed.

For samples that are collected for metals analyses, or other analysis not affected by the silastic tubing, it is permissible to collect the sample directly from the discharge of the pump head tubing after an adequate purge has been completed. When collecting samples in this manner, there are several considerations to be aware of. The pump head tubing (silastic, etc.) must be changed after each sample and a rinsate blank must be collected from a representative piece of the pump head tubing (only one blank per investigation). Also, precautions must be taken to ensure that the end of the discharge tubing is not allowed to touch the ground, or other surface, in order to maintain the integrity of the sample when it is collected in this manner.

11.3 Syringe

Syringes, in conjunction with PushPoint® samplers, can be used to collect pore water samples if the integrity of the sample analysis will not be compromised. The tubing from the sampling port of the PushPoint® can be directly attached to a syringe and a pore water sample can be manually withdrawn from the sediment. The syringe can be used as the final sample container or the pore water can be transferred to another container, depending on project objectives and analytical requirements.

11.4 Sample Handling and Preservation Requirements

1. Pore water will typically be collected from sediments using a PushPoint® and placed directly into the sampling containers. A syringe may then be used to transfer the sample from the sampling container into the appropriate container.

2. When transferring the pore water sample from a collection device, make sure that the device does not come in contact with the final sample containers. The syringe used in the sample transfer is the only piece of equipment that should be in contact with the transfer vessel and the final sample container.

3. Place the sample into the appropriate labeled container. Samples collected for VOC

analysis must not have any headspace (see Section 7.1). All other sample containers must be filled with an allowance for ullage.

4. All samples requiring preservation must be preserved as soon as practically possible after sample collection. If preserved VOA vials are used, these will be preserved with concentrated hydrochloric acid prior to departure for the field investigation. All other chemical preservatives required for the remaining suite of analytes will be specified in the site-specific SAP. The adequacy of sample preservation will be checked after the addition of the preservative for all samples, except for the samples collected for VOC analysis. If it is determined that a sample is not acceptably preserved, additional preservative should be added to achieve adequate preservation. Preservation requirements for surface water samples will be specified in the site-specific SAP/QAPP and the Sample Preservation SOP FLD 03.00.

12.0 DATA RECORDS AND MANAGEMENT

Once collected, samples are preserved, labeled, and stored for transport. A chain of custody form must accompany all samples during transport and transfer between entities. Sample labels should contain the following information:

- Site identification
- Date sampled
- Location identification
- Sampler initials
- Time
- Analysis to be performed
- Preservative

Any other pertinent data should be recorded in the site dedicated field logbook.

13.0 QUALITY CONTROL AND ASSURANCE

The following general QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. In general, concurrent (duplicate) sample collection at a frequency of 10% is required for most sampling activities. Field blanks at a frequency of one per day are also generally required. Consult the corresponding SAP/QAPP for specific QA/QC sampling frequency. Below is a list of usual pore water QA/QC sample types and the inaccuracy they are intended to detect:

   - Duplicate sample – two samples collected at the same location at the same time, intended to detect variability inherent in collection, processing, and handling procedures
   - Field blank – checks cross-contamination during sample collection, preservation, and shipment as well as in the laboratory.
   - Rinsate blank – detects equipment contamination due to inadequate decontamination

procedures

3. All instrumentation should be operated in accordance with operating instructions as supplied by the manufacturer unless otherwise specified in the work plan or SAP/QAPP. Equipment calibration activities should be conducted and documented prior to sampling and/or operation of equipment.
4. Document any deviations from SOPs, work plan, SAP/QAPP, etc.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

MHE Products, PushPoint® Sampler (US Pat. # 6,470,967), Operators Manual and Applications Guide, Ver. 2.01, 2/15.

15.0 FIGURES

Figures 1-4 show equipment operation and basic sampling techniques.

Figure 1 - Pore Water PushPoint®

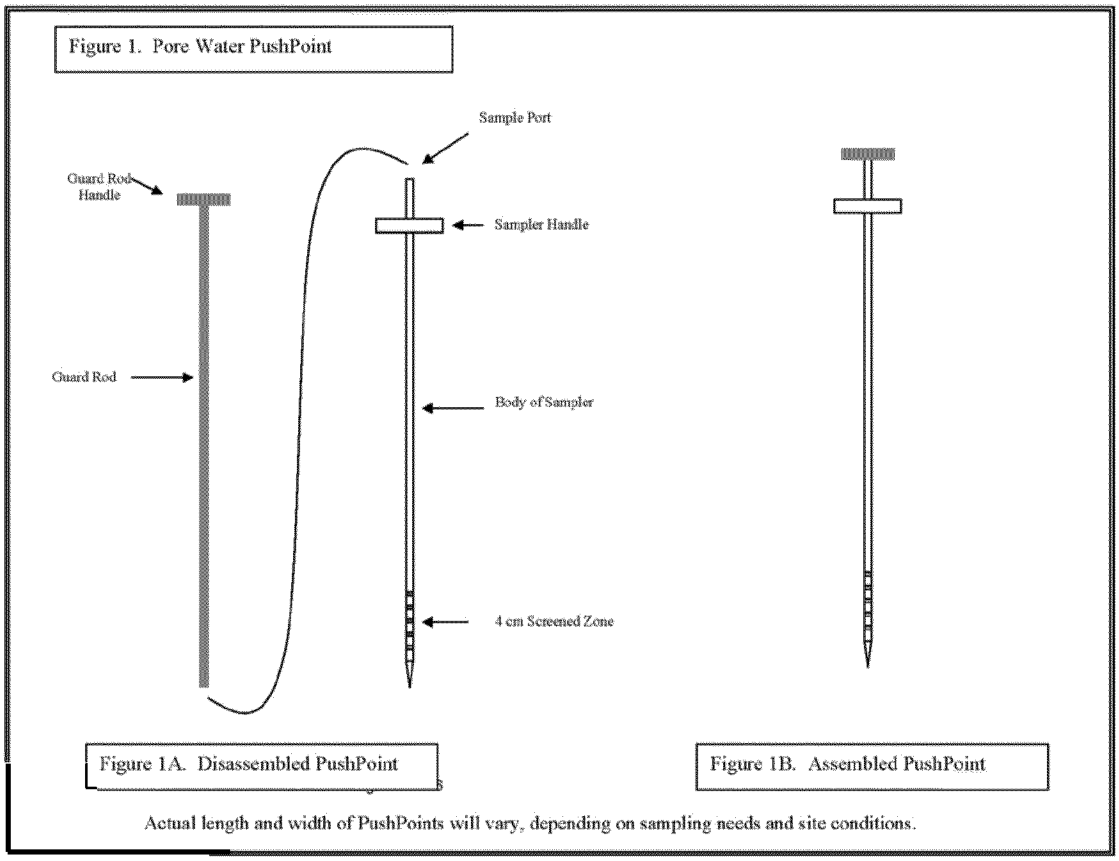


Figure 2 - PushPoint® deployed with a Sampling Platform

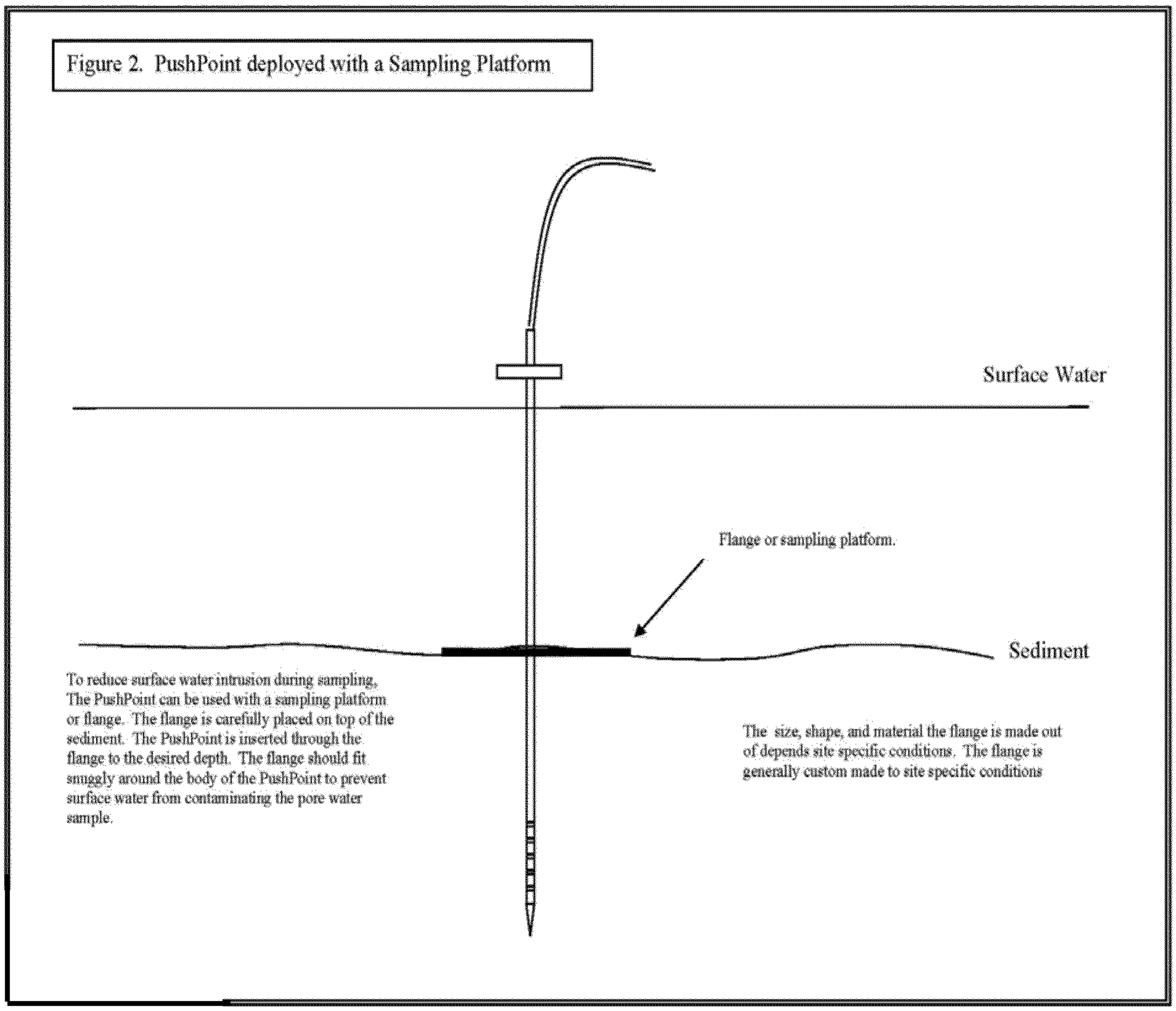


Figure 3 - PushPoint® Being Deployed into the Sediment

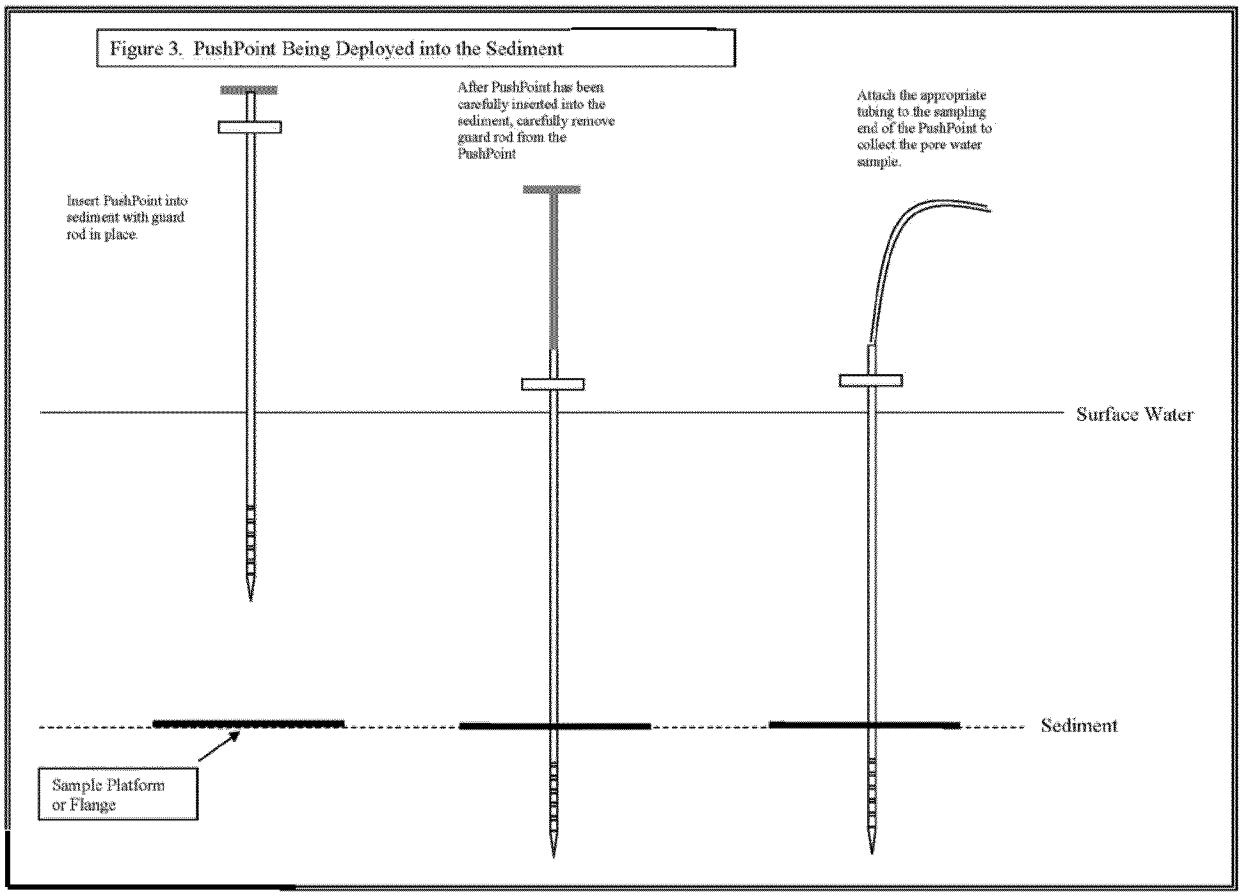
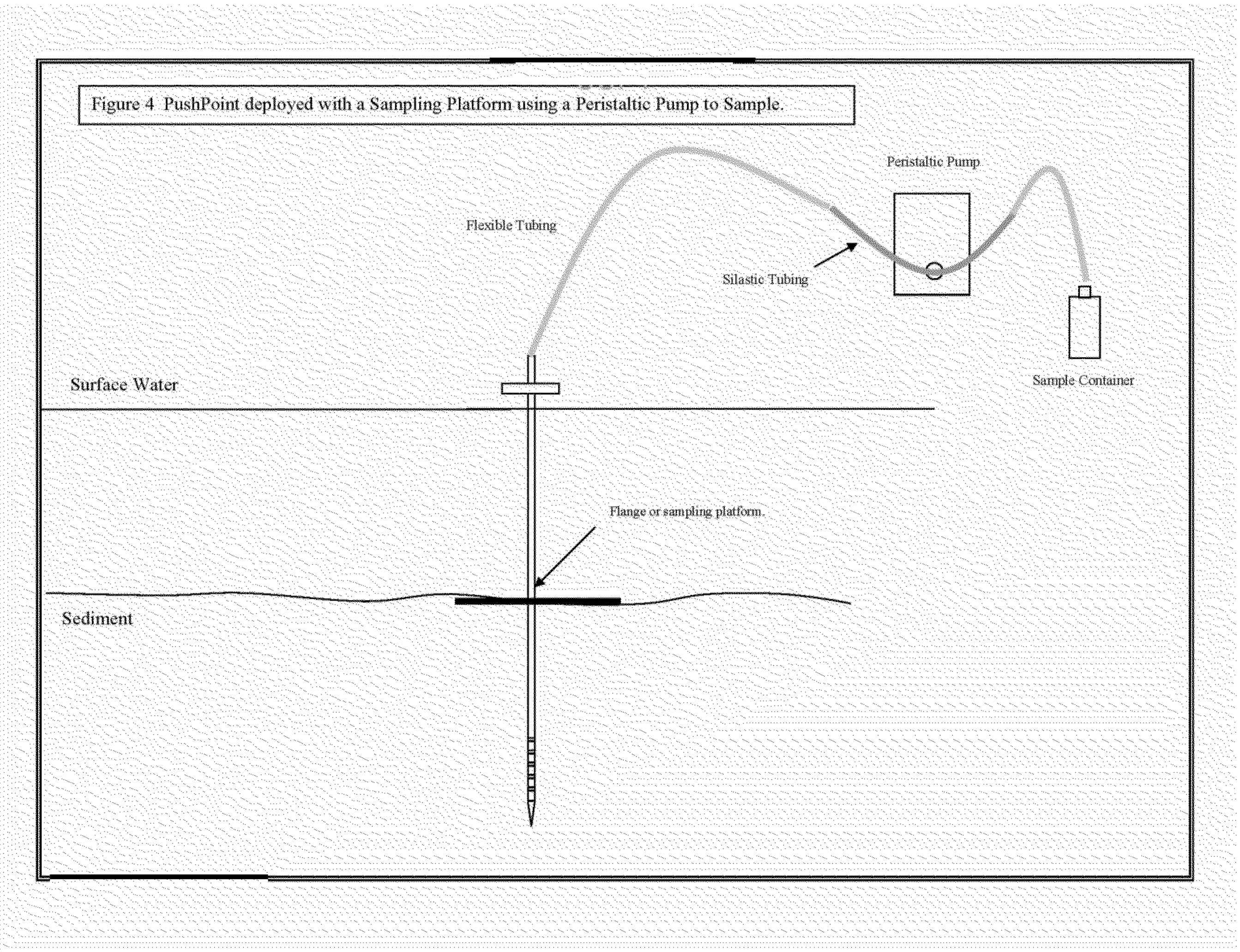


Figure 4 - PushPoint® deployed with a Sampling Platform using a Peristaltic Pump to Sample



Sample Custody and Labeling

APPROVED:

ESAT Region 8 QA Coordinator — Date: 04/04/12

ESAT Region 8 Team Manager — Date: 6/6/12

EPA Task Order Project Officer — Date: 7/10/12

ESAT Region 8 Task Lead — Date: 6/8/12

DCN: EP8-7-7061

This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to assist field personnel in developing proper sample custody and sample identification methods for the collection of environmental samples. This includes the use of chain of custody (COC) forms and labels for samples collected in the field. These procedures are critical in ensuring the integrity of environmental samples.

2.0 SCOPE AND APPLICABILITY

To ensure the integrity of a sample collected in the field or generated in a laboratory setting, documentation is needed to chronicle all sample handling for collection or creation through analysis and/or disposal. Any sample that is collected in the field or generated in a laboratory setting will require that records are kept as it transfers from various entities. This is the basis for generation of a COC. Uniquely, labeling samples with information, such as sample location, date, time, preservation method, and analytical requirements, keeps samples organized. A COC is initiated for each sample, either at the time of sample collection or generation or as part of preparation for a sampling event. This SOP will cover the best practices for sample custody and the method of COC and label generation.

3.0 SUMMARY OF METHOD

Once a sample is collected, several steps need to be taken to ensure the required information is collected and maintained as it is transferred from the point of collection to the laboratory. If sample nomenclature and location is known before a field event, a COC will be generated before deployment into the field. When generating the COC, it is important to know the analytical fate of samples required for each sample location (e.g. total recoverable metal, dissolved metals, etc.). This information can be found in the site-specific Sampling and Analysis Plan (SAP) and other sampling event planning documents. Some software programs (e.g. Scribe) that generate COCs also have the ability to generate labels. Scribe is the Laboratory Information Management System (LIMS) used by the lab. It is important to keep in mind that it is not mandatory to generate COCs and labels before a sampling event, but it is preferred. If it is not known where samples will be collected or the nomenclature of the sites is unclear, sample containers can be labeled with permanent marker with tape placed over it, and a blank COC can be filled out at the time of sample collection. Once the method of custody is established, a specific person, known as the sample custodian, is then responsible for maintaining the integrity of the samples as they move from and within various locations.

4.0 ACRONYMS AND DEFINITIONS

CLP	Contract Lab Program
COC	Chain of Custody
EPA	United States Environmental Protection Agency
ERT	Environmental Response Team
ID	Identification
LIMS	Laboratory Information Management System
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure

Chain of Custody (COC): A document used to chronologically track movement of samples between entities from collection to disposal.

Sampling and Analysis Plan (SAP): A site-specific document that describes the events to take place in the field.

Scribe: – A software tool developed by the United States Environmental Protections Agency (EPA) Environmental Response Team (ERT) to assist in the process of managing environmental data. Scribe captures sampling, observational, and monitoring field data.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

5.0 HEALTH AND SAFETY

There are no specific health and safety hazards associated with sample custody and labeling, but these activities sometimes take place on-site during a sampling event. It is important for field personnel to familiarize themselves with the site-specific Health and Safety Plan before deployment to a site. In terms of personal interaction with the sample throughout the process of sample custody, there exists the possibility that the samples can leak. It is important to be aware of such hazards, especially when interacting with samples that are highly contaminated.

6.0 CAUTIONS

Samples sometimes require specific storage and maintenance, such as temperature preservation requirements. Proper storage of samples is critical in maintaining their integrity. Labeling is also critical in the process of sample custody. Samples usually are labeled with a series of letters and numbers that correspond to a site location, which sometimes are very similar to each other. Sample nomenclature will be designated in the approved SAP and will be followed in the field. Once a COC or label is generated, it is very important to have it reviewed for quality assurance purposes. Sample label and COC review is necessary to ensure that they match site documents.

7.0 INTERFERENCES

Once a COC and group of labels are reviewed and deployed, it is critical that the proper label ends up on the correct sample container. There will be more than one subsample collected at the majority of sampling locations in the region. This means that sample numbers can be very close in nomenclature, which puts more emphasis on attention to detail when labeling the sample containers. If the wrong label is attached to a sample, it may result in improper preservation, improper analysis, or rejection by the analytical laboratory.

8.0 PERSONNEL QUALIFICATIONS

It is critical that field personnel have proper clearance and health and safety training. Anyone who performs sample custody activities should also familiarize themselves the site-specific SAP and Quality Assurance Project Plan (QAPP), as well as with applicable SOPs: Surface Water Sampling SOP FLD 1.00, Groundwater Sampling SOP FLD 04.00, Soil Sampling SOP FLD 5.00, Pore Water Sampling SOP FLD 10.00, and Shallow Stream Sediment Sampling SOP FLD 06.00.

9.0 EQUIPMENT AND SUPPLIES

Below is a list of equipment and supplies required for COC activities (refer to the site specific SAP for additional items that maybe needed:

- Scribe software
- A SAP that details sample locations and analytical requirements
- Printer (that accepts corresponding labels)
- Blank COC pages in case of unexpected opportunistic sampling
- Permanent marker for preliminary labeling
- Clear tape for label protection from moisture
- Printable labels
- Field Logbook

10.0 STANDARDS AND REAGENTS

There are no standards or reagents associated with this SOP.

11.0 PROCEDURES

The following sections outline the general procedures for sample custody and labeling, filling out COCs with the proper information, and relinquishing samples. See Attachment A for an example of a blank COC and Attachment B for an example of a sample label.

11.1 Generating a Blank COC and Sample Labels

There are several types of data management software that can be used to generate COCs and labels. Scribe is used at the EPA Region 8 laboratory. Some training is required before an individual can use Scribe; however, once the basics of Scribe are understood, it can be used to generate COCs and labels for any type of sample or analysis. A COC that is generated prior to deployment should have the following information:

- Site Identification
- Analysis to be performed
- Preservation
- Tag Identification

The following information should not be filled out until sampling occurs:

- Date
- Time
- Sampler identification
- Comments describing anomalies

Labels can be produced with the same information found in the COCs.

11.2 Populating COC Fields and Affixing Labels

Sample containers should always be marked with a permanent marker with the site identification (ID), time of collection, analysis to be performed, date, and sampler initials prior to sample collection. Once samples have been collected, and a safe place to fill out COC and labels is established, field personnel should fill out the pre-populated COCs and labels with information such as date, time of collection, sampler initials, and comments. It is imperative that the information written on the sample container in permanent marker is the same information on the sample labels and the COC. The same information should also be recorded in a site-dedicated field logbook.

Once the labels have been verified to have the correct information, they should be affixed to the sample containers. Always be sure to double check that the proper label is placed on the corresponding sample container by cross-referencing it with the markings. Once the label is affixed to the sample container, place clear packing tape over the label and wrap completely around the container. This will prevent moisture from dissolving the label adhesive and blurring the writing. It also prevents holes, knicks, or tears from rendering the label unreadable.

11.3 Review/Custody Transfer

Once sample information is written on the COC and labels, and the label IDs have been verified against the permanent marker ID on the container, they are then ready for transfer of custody. Whether the samples are going to the EPA Region 8 lab or a Contract Lab Program (CLP) laboratory partner, the samples must be properly shipped at the required temperature (4°C for water and sediment samples) and done so in a way that containers are not compromised. In order to not compromise the integrity of the samples, the handler needs to make sure the cooler or other transporting vessel is not dropped, exposed to moisture or extreme weather, or in any other way disturbed. A signed copy of the COC intended for the receiving laboratory (samples IDs and event information should not be viewable to the lab) must be included in the shipping container. If samples are returning to the Region 8 Laboratory, they should be properly stored on ice in the field until delivered to the lab. To protect against sample contamination, place the ice in the coolers in plastic bags. When at the lab, samples should be placed in the walk-in coolers located in the sample receiving room. A signed copy of the COC is given to the sample receiving coordinator. In order to ensure samples are transferred to the correct party with the appropriate information and communication, a mutual signing of the COC by the sampler or transport agency and the sample coordinator can be arranged.

12.0 DATA RECORDS AND MANAGEMENT

As mentioned earlier, a COC should have information such as site ID, sample location, sample time, sample date, sampler initials, analytical requirements, sample matrix, preservative type, and a comments field. A sample label should have information such as sample location, time, date, matrix, preservative, and sampler initials. Any other field observations that require an explanation should be noted in the field forms or site-dedicated field notebook. Data such as sample ID, time, date, field parameters, (pH, temperature, conductivity, and dissolved oxygen) and sampler initials will eventually be entered into Scribe.

13.0 QUALITY CONTROL AND ASSURANCE

Proper sample custody and labeling requires a number of quality control and assurance steps. A COC generated in Scribe should always be crossed-checked by another person with the sample list found in the SAP. Completed COCs and labels should also be compared for accuracy before being relinquished to the receiving analytical laboratory. Any incorrect information on a COC or label may cause the lab to reject the shipment.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

15.0 ATTACHMENTS

Attachment A: Example Chain of Custody Form

TechLaw
ESAT Region 8 Laboratory
16194 W 45th Drive
Golden, CO 80403
303.312.7047

US EPA CLP
Chain-of-Custody

Page 1 of 3

EVENT: 2011_eCOC Template

Sample #	Tag	Location	Sub Location	Sample Type	Collection	Matrix	Analyses	Preservation	Sample Date	Sample Time	Sampler	Remarks

Relinquished By (DATE):

Relinquished By:

Received By (DATE/TIME):

Received By:

Cooler Temp:________
ICE: Y N
pH: Y N
Cust. Seals: Y N
COC/Labels Agree: Y N
Containers Intact: Y N

Attachment B: Example Sample Label

Sample # 082X-127 **Sampler:**
Tag: A
Date: Sample Time:
Location: Dup-05 Samp_Depth:
Analyses: Total Recoverable Metals
Preservation: TR_Plastic Baggie

General Field Sampling Protocols

APPROVED:

ESAT Region 8 QA Coordinator — Date: 04/04/12

ESAT Region 8 Team Manager — Date: 6/6/12

EPA Task Order Project Officer — Date: 7/10/12

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide general field sampling guidelines that will assist Environmental Protection Agency (EPA) and Environmental Services Assistance Team (ESAT) personnel in choosing sampling strategies, location, and frequency for proper assessment of site characteristics. This SOP is applicable to all field activities that involve sampling.

2.0 SCOPE AND APPLICABILITY

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

3.0 SUMMARY OF METHOD

Sampling is the selection of a representative portion of a larger population, area or body. Through examination of a sample, the characteristics of the larger entity from which the sample was drawn can be inferred. In this manner, sampling can be a valuable tool for determining the presence, type, and extent of contamination by hazardous substances in the environment. The sampling design is a fundamental part of data collection for scientifically based decision making. A well-developed sampling design plays a critical role in ensuring that data are sufficient to draw the conclusions needed. The goals of a sampling design can vary widely. Typical objectives of a sampling design for environmental data collection are:

- To support a decision about whether contamination levels exceed a threshold of unacceptable risk
- To determine whether certain characteristics of two populations differ by some amount
- To estimate the mean characteristics of a population or the proportion of a population that has certain characteristics of interest
- To identify the location of “hot spots” (areas having high levels of contamination) or plume delineation
- To characterize the nature and extent of contamination at a site
- To monitor trends in environmental conditions or indicators of health

A well-planned sampling design is intended to ensure that resulting data are adequately representative of the target population and defensible for their intended use. Representativeness may be considered as the measure of the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Throughout the sampling design process, the efficient use of time, money, and human resources are critical considerations. A good design should meet the needs of the study with a minimum expenditure of resources. If resources

4.0 ACRONYMS AND DEFINITIONS

EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
DOT	Department of Transportation

HAZWOPER	Hazardous Waste Operations and Emergency Response
IATA	International Air Transport Association
MI	Multi-increment
OSHA	Occupational Safety and Health Administration
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure

Occupational Safety and Health Administration (OSHA): A regulatory agency that governs health and safety standards in the United States.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

Quality Assurance Project Plan (QAPP): A site-specific document that specifies quality assurance activities and data quality objectives.

5.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. Environmental Protection Agency (EPA), Occupational Safety and Health Administration (OSHA) and corporate health and safety procedures. Always review the site Health and Safety Plan (HASP) before beginning work at any site.

6.0 CAUTIONS

In general, health and safety of field team members and sample/data integrity are the two main concerns during a field sampling event. Field personnel must understand sampling procedures and be familiar with health and safety protocols before deployment to a site. Always consult the HASP before entering a site.

7.0 INTERFERENCES

The nature of the object or materials being sampled may be challenging to characterize. If a material is homogeneous, it will generally have a uniform composition throughout. In this case, any sample increment can be considered representative of the material. On the other hand, heterogeneous samples present problems to the sampler because of spatial and temporal changes in the material. Samples of hazardous materials may pose a safety threat to both field and laboratory personnel. Proper health and safety precautions should be implemented when handling this type of sample. Environmental conditions, weather conditions, or non-target chemicals may cause problems and/or interferences when performing sampling activities or when sampling for a specific parameter. Refer to the specific SOPs for sampling techniques.

8.0 PERSONNEL QUALIFICATIONS

All personnel who participate in field activities are required to obtain clearance in three mandatory health and safety programs: medical monitoring, respirator fit testing, and OSHA Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. In addition, any personnel who will participate in sampling activities must read, understand, and sign the site-specific HASP and associated Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPP).

9.0 EQUIPMENT AND SUPPLIES

The equipment required to collect samples must be determined on a site-specific basis. Due to the wide variety of sampling equipment available, refer to the specific SOPs for sampling techniques which include lists of the equipment required for sampling.

10.0 STANDARDS AND REAGENTS

Reagents may be utilized for preservation of samples and for decontamination of sampling equipment. The preservatives required are specified by the analysis to be performed. Decontamination solutions are specified in the Sampling Equipment Decontamination SOP FLD 02.00.

11.0 PROCEDURES

11.1 Types of Samples

In relation to the media to be sampled, two basic types of samples can be considered: the environmental sample and the hazardous sample.

Environmental samples are those collected from streams, ponds, lakes, wells, and are off-site samples that are not expected to be contaminated with high levels of hazardous materials. They usually do not require the special handling procedures typically used for concentrated wastes. However, in certain instances, environmental samples can contain elevated concentrations of pollutants and in such cases would have to be handled as hazardous samples.

Hazardous or concentrated samples are those collected from drums, tanks, lagoons, pits, waste piles, fresh spills, or areas previously identified as contaminated, and require special handling procedures because of their potential toxicity or hazard. These samples can be further subdivided based on their degree of hazard; however, care should be taken when handling and shipping any wastes believed to be concentrated regardless of the degree. The importance of making the distinction between environmental and hazardous samples is two-fold:

1. Personnel safety requirements: Any sample thought to contain enough hazardous materials to pose a safety threat should be designated as hazardous and handled in a manner which ensures the safety of both field and laboratory personnel. Personnel handling potentially hazardous substances should always wear proper Personal Protective Equipment.

2. Transportation requirements: Hazardous samples must be packaged, labeled, and shipped according to the International Air Transport Association (IATA) Dangerous Goods Regulations or Department of Transportation (DOT) regulations and U.S. EPA guidelines.

11.2 Sample Collection Techniques

In general, two basic types of sample collection techniques are recognized, both of which can be used for either environmental or hazardous samples.

Grab (Discrete) Samples
A grab sample is defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected all at once at one particular point in the sample medium. The representativeness of such samples is defined by the nature of the materials being sampled. In general, as sources vary over time and distance, the representativeness of grab samples will decrease.

Composite (Multi-Increment) Samples
Multi-increment (MI) or composite sampling is a structured sampling protocol that reduces data variability and increases sample representativeness. The objective of MI sampling is to obtain a single sample for analysis that has a mean analyte concentration representative of the decision unit. The decision unit size is site-specific and represents the smallest area on which to base a decision or conclusion. Samples are collected from multiple locations within the decision unit and composited so the samples are spatially representative of the decision unit. The decision unit must be defined so that the results are relevant to explicitly articulated sampling objectives. Note that establishment of decision units is necessary to develop any effective sampling approach, whether using MI or discrete sampling.

The MI sampling strategy improves the reliability and defensibility of sampling data by reducing their variability compared to conventional discrete sampling strategies. The data distribution for MI replicate samples tends to be normally distributed, as contrasted to the positively skewed distribution seen with discrete samples. Fewer non-detect results can be expected using MI, thus mitigating problems caused by using censored data sets and lessening the chance of missing significant contamination. In addition, levels of statistical confidence and decision uncertainty that would require a large number

11.3 Types of Sampling Strategies

It is important to select an appropriate sampling approach for accurate characterization of site conditions. Prior to undertaking any sampling program, it is necessary to establish appropriate measurement and system Data Quality Objectives. Refer to the U.S. Environmental Protection Agency (EPA) Soil Sampling Quality Assurance User's Guide (listed in Section 14.0 References) for guidance in establishing Data Quality Objectives, statistical sampling methodologies and protocols for each of the sampling approaches. Each approach is defined below.

Judgmental or Biased Sampling
Judgmental or Biased sampling is used primarily for documenting an observed release to the groundwater, surface water, air or soil exposure pathways. This form of sampling is based on the subjective selection of sampling locations where contamination is most likely to occur. Locations are based on relative historical site information and on-site investigation (site walk-over) where contamination is most likely to occur.

There is no randomization associated with this sampling approach because samples are primarily collected at areas of suspected highest contaminant concentrations. Any statistical calculations based on the results of this sampling technique will be biased.

Random Sampling

Random sampling, used for the characterization of a heterogeneous non-stratified waste, involves arbitrary collection of samples within a defined area. This method is most effective and accurate if the chemical heterogeneity of the waste remains constant from batch to batch. The easiest method for Random Sampling is to divide the area for sampling into an imaginary grid, assign a series of numbers to the units of the grid, and select the numbers or units to be sampled through the use of a random-numbers table which can be found in the text of any basic statistics book. Note that haphazardly selecting sample numbers or units is not a suitable substitute for a randomly selected sample.

Stratified Random Sampling

Stratified random sampling, used for the characterization of a heterogeneous stratified waste, involves arbitrary collection of samples within a defined area and strata. This method is most effective and accurate if the chemical heterogeneity of the waste remains constant from batch to batch. The easiest method for stratified random sampling is to divide the area for sampling into an imaginary grid, assign a series of numbers to the units of the grid, and select the numbers or units to be sampled through the use of a random-numbers table which can be found in the text of any basic statistics book. A random sample is then collected from each strata at the selected numbers or units on the grid. Note that haphazardly selecting sample numbers or units is not a suitable substitute for a randomly selected sample.

Systematic Grid Sampling

Systematic grid sampling involves dividing the area of concern into smaller sampling areas using a square or triangular grid. Samples are then collected from the intersection of the grid lines or nodes. The origin and direction for placement of the grid should be selected by using an initial random point. The distance between nodes is dependent upon the size of the site or area of concern and the number of samples to be collected. Generally, a larger distance is used for a large area of concern.

Systematic Random Sampling

Systematic random sampling involves dividing the area of concern into smaller sampling areas. Samples are collected within each individual grid cell using random selection procedures.

Search Sampling

Search sampling utilizes a systematic grid or systematic random sampling approach to define areas where contaminants exceed clean-up criteria. The distance between the grid lines and number of samples to be collected are dependent upon the acceptable level of error (i.e., the chance of missing a hot spot). This sampling approach requires that assumptions be made regarding the size, shape, and depth of hot spots.

Transect Sampling

Transect sampling involves establishing one or more transect lines, parallel or non-parallel, across the area of concern. If the lines are parallel, this sampling approach is similar to systematic grid sampling. The advantage of transect sampling over systematic grid sampling is the relative ease of establishing and relocation transect lines versus an entire grid. Samples are collected at regular intervals along the transect line at the surface and/or at a specified depth(s). The distance between the sample locations is determined by the length of the line and the number of samples to be collected.

11.4 Quality Assurance Project Plans (QAPP)

A Quality Assurance Project Plan (EPA, 2006) is required when it becomes evident that a field investigation is necessary. It should be initiated in conjunction with, or immediately following, notification of the field investigation. This plan should be clear and concise and should detail the following basic components, with regard to sampling activities:

- Objective and purpose of the investigation
- Basis upon which data will be evaluated
- Information known about the site including location, type and size of the facility, and length of operations/abandonment
- Type and volume of contaminated material, contaminants of concern (including concentration), and basis of the information/data
- Technical approach including media/matrix to be sampled, sampling equipment to be used, sample equipment decontamination (if necessary), sampling design and rationale, and SOPs or description of the procedure to be implemented
- Project management and reporting, schedule, project organization and responsibilities, manpower and cost projections, and required deliverables
- QA objectives and protocols including tables summarizing field sampling and QA/QC analysis and objectives

Note that this list of QAPP components is not all-inclusive and that additional element(s) may be added or altered depending on the specific requirements of the field investigation. It should also be recognized that although a detailed QAPP is quite important, it may be impractical in some instances. Emergency responses and accidental spills are prime examples of such instances where time might prohibit the development of site-specific QAPPs prior to field activities. In such cases, investigators would have to rely on general guidelines and personal judgment, and the sampling or response plans might simply be a strategy based on preliminary information and finalized on site. In any event, a plan of action should be developed, no matter how concise or informal, to aid investigators in maintaining a logical and consistent order to the implementation of their task.

11.5 Legal Implications

The data derived from sampling activities are often introduced as critical evidence during litigation of a hazardous waste site cleanup. Legal issues in which sampling data are important may include cleanup cost recovery, identification of pollution sources and responsible parties, and technical validation of remedial design methodologies. Because of the potential for involvement in legal actions, strict adherence to technical and administrative SOPs is essential during both the development and implementation of sampling activities.

Technically valid sampling begins with thorough planning and continues through the sample collection and analytical procedures. Administrative requirements involve thorough, accurate documentation of all sampling activities. Documentation requirements include maintenance of a chain of custody, as well as accurate records of field activities and analytical instructions. Failure to observe these procedures fully and consistently may result in data that are questionable, invalid and non-defensible in court, and the consequent loss of enforcement proceedings.

12.0 DATA RECORDS AND MANAGEMENT

There are many data parameters and custody records that require attention to detail. Refer to the specific SOPs for data management activities that are associated with sampling techniques.

13.0 QUALITY CONTROL/QUALITY ASSURANCE (QC/QA)

Refer to the specific SOPs for the type and frequency of QA/QC samples to be analyzed, the acceptance criteria for the QA/QC samples, and any other QA/QC activities which are associated with sampling techniques.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6. April 2007.
EPA Guidance on Systematic Planning using the Data Quality Objectives Process (QA/G-4). February 2006.

EPA Guidance on Choosing a Sampling Design for Environmental Data Collection (QA/G-5S). December, 2002

COLLECTION, ANALYSIS AND DISPOSAL OF ESAT LABORATORY WASTE

APPROVAL PAGE

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ESAT Review: 1/17/2007
ESAT QA/QC Coordinator — Date

ESAT Approval: 1/17/2007
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1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure describes the procedures and practices for safely collecting, analyzing and storing aqueous corrosive wastes.

1.2 This procedure is applicable to the following aqueous waste streams; acidic instrument waste, acidic reagents (except standards), sample digestates and samples preserved for analysis with acid.

1.3 This procedure is applicable to solid wastes such as soils, vegetation and biota samples.

2.0 SUMMARY OF PROCEDURE

2.1 Aqueous sample, digestates, reagents and some instrument waste contain small amounts of mineral acids.

2.1.1 The presence of these acids causes the pH of the waste to be below 2 and hence be defined as hazardous.

2.1.2 In addition, these wastes may contain metal concentrations which exceed discharge standards.

2.2 Solid wastes may contain metal concentrations which exceed disposal standards.

2.2 This waste must be properly labeled, contained and stored in accordance with all state, federal and ESAT regulations.

2.2.1 This procedure includes the initiation of satellite waste containers, documentation accompanying the waste and procedures for placing the waste in the designated waste storage area.

3.0 DEFINITIONS / ACRONYMS

3.1 Aqueous Corrosive Waste - An aqueous solution with a pH<2.

3.2 Evidentiary Materials – Samples, sample containers and sample residuals.

3.3 F Waste: Hazardous waste from nonspecific sources (See 40 CFR 261.31.).

3.4 Satellite Waste Container - A container used to collect waste during generation.

3.5 Secondary Containment

3.5.1 A second level of containment ensuring no release if the initial containment fails.

3.6 Waste Control Officer (WCO)

3.6.1 Person responsible for maintaining control of all documentation of waste disposal.

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4.0 HEALTH AND SAFETY

4.1 All pertinent procedures outlined in the EPA Region 8 Chemical Hygiene Plan (CHP) will be followed in performance of the handling of wastes.

4.1.1 The use of laboratory equipment and chemicals exposes the analyst to several potential hazards.

4.1.2 Good laboratory technique and safety practices should be followed at all times.

4.2 Solutions classified as aqueous corrosive wastes normally contain percentage levels of mineral acids and can contain certain inorganic elements known to be hazardous.

4.2.1 Gloves, protective eye wear and laboratory coats should be worn at all times when handling samples, reagents, or when in the vicinity of others handling these items.

4.3 Satellite waste containers must always be tightly capped when not in use.

4.4 Satellite waste containers can weigh in excess of 50 lbs and should be lifted carefully.

4.5 Spilled samples, reagents, and water should be cleaned up from instrument and autosampler surfaces immediately.

4.5.1 In the case of acid spills, the acid should be neutralized with acid spill kits available in the laboratory.

5.0 CAUTIONS

5.1 Prior to starting work that involves handling wastes, personnel should review the project plan safety requirements, analytical procedure safety requirements, and this waste management procedure.

5.1.1 When in doubt as to the proper procedure to follow, contact the Health and Safety Officer for guidance.

5.1.2 Personnel should minimize exposure to potential health hazards through the use of engineering and administrative controls, work practice procedures and practices, and proper protective equipment.

6.0 PERSONNEL QUALIFICATIONS

6.1 Personnel Responsibilities

6.1.1 Personnel responsibilities for hazardous waste management at EPA Region 8 laboratory are described in the Introduction/Executive Summary of the *Safety, Health and Environmental Management Program (SHEMP) Manual* (Section 2.2 Hazardous Waste Management).

6.2 Personnel Training

6.2.1 Federal and state regulations require that employees who handle hazardous waste be provided with initial and annual training. Initial orientation and on-the-job training are provided to new ESAT employees within their first month of employment, and refresher training is provided on an annual basis thereafter.

6.2.2 This training is designed to keep employees familiar with waste handling procedures in place at EPA, along with applicable regulations.

6.2.3 Training completion will be enforced by the supervisor and documented for each

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individual by the ESAT Health and Safety Officer.

7.0 EQUIPMENT AND SUPPLIES

7.1 The WCO (Waste Control Officer) will assure that a supply of appropriate waste containers and labels are available for use.

7.2 Waste containers must be able to be tightly capped and both the container and the secondary containment must be chemically resistant to corrosive materials.

8.0 SATELLITE WASTE CONTAINER PREPARATION

8.1 The waste container will be properly labeled as appropriate.
- 8.1.1 A Waste Container ID is assigned to the container by the person who initiates the container's designation.
  - 8.1.1.1 The container ID will be written on the container with a Sharpie in such a location as to be easily seen.
- 8.1.2 The Container ID will follow the YYMMDD-XXXX-# format where "XXXX" describes the laboratory room number and the "#" will be the number of the container generated on that particular day.
- 8.1.3 Affix an appropriate red and white hazardous label for containers used for suspected or known hazardous materials.
- 8.1.4 Affix a black and white label warning of the presence of corrosives (pH<2) when the contents to be added to the container are known to be acidic.

8.2 A waste container inventory log will be initiated and placed next to the container.
- 8.2.1 An attached (or located in the near vicinity) hazardous waste container inventory log, listing the accumulated waste maintained by the generator(s).
- 8.2.2 The waste inventory sheet should include the container ID number and its date, description and amount of waste added, date of the addition, and the name of the person making each addition.

Note: The waste inventory sheet serves two important purposes. It guards against addition of incompatible chemicals to the container mix and allows packers to determine the correct classification of the waste for transport and disposal.

9.0 WASTE COLLECTION AND ANALYSIS

9.1 Waste must be collected as near as possible to the point of generation and have secondary containment.

9.2 Containers must be kept closed except when waste is being added.

9.3 When a container of waste is approximately 85% full, the waste inventory sheet must be signed, dated and entered into the inventory system by the WCO.

9.4 Full aqueous waste containers will be sub-sampled, the waste inventory sheet filled out and transported to the waste storage area within 24 hours.
- 9.4.1 The waste inventory sheet must be clearly labeled as "Awaiting analysis".

9.5 Waste containers that are full or otherwise ready for disposal will be transported to the F wing where the wastes are segregated by waste categories: D00I, ignitable liquids; D002, corrosive materials; D003, reactive materials; and D004 to D043, toxic materials.
9.5.1 Each unit will be labeled with the start of accumulation date and, as appropriate, a "hazardous waste" or "non-hazardous waste" label.

9.6 Analysis of each aqueous waste container must be completed within 10 days.
9.6.1 The results are then entered on the waste inventory sheet.

9.7 Solid wastes designated for disposal are collected in an approved container.
9.7.1 In lieu of analyzing the solids, the data collected during the analysis of the samples is used to indicate the level, if any, of metals concentration in the solid waste.
9.7.2 In general, segregating the solids into LIMS workorder specific groups will make calculating the metals concentration less complicated.

9.8 A copy of the completed waste inventory sheet along with the raw data is provided to the WCO and the original is attached to the waste container.

10.0 DATA AND RECORDS MANAGEMENT

10.1 At EPA Region 8 laboratory, waste is tracked through the use of container labels, waste container logs, an in-house tracking system, physical inventories, hazardous waste shipping manifests, and certificates of disposal.

10.2 Data from waste container sheets for wastes generated in the laboratory is entered into the Hazardous Waste Tracking System (HWTS) bound green notebook by the WCO, at the time of transport to the F wing.

11.0 WASTE MINIMIZATION

11.1 Metals laden waste volumes are minimized by the use of a dedicated waste receptacle in which no other laboratory waste is placed.
11.1.1 Waste concentrations are minimized by judicious use of metals standard solutions and materials.
11.1.2 In addition, ESAT chemists are working with field personnel to reduce the amount of excess sample collected.

12.0 REFERENCES

12.1 EPA Region 8, Chemical Hygiene Plan, current version.

12.2 EPA Region 8, Health and Safety Plan, current version.

12.3 EPA Region 8, Waste Management SOP, current version.

SAMPLE RECEIPT, CUSTODY, STORAGE AND LIMS ENTRY OF SAMPLES

APPROVAL PAGE

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1.0 SCOPE AND APPLICABILITY

1.1 The purpose of this standard operating procedure (SOP) is to establish a safe, traceable, and consistent laboratory process for receiving, tracking, and storage of ESAT samples at the USEPA Region 8 Laboratory. These may include surface waters, ground waters, soils, sediments and biological materials.

2.0 SUMMARY OF PROCEDURE

2.1 This SOP specifies the requirements for project definition, sample receipt, control, and record keeping by ESAT in the USEPA Region 8 Laboratory. The following objectives are defined in detail within this document:

2.2 EPA Project Definition - Prior to accepting samples at the laboratory, an agreement between the EPA Project Officer (PO) and the laboratory must be set forth in a Technical Direction Form (TDF) which details the required analytical methods, target analytes, approximate quantity of samples, receipt date, analytical quality control procedures and data deliverables.

2.3 ESAT Project Definition - Following receipt of the TDF, ESAT personnel will create a project in LIMS that defines the requirements detailed in the TDF.

2.4 Sample Integrity Inspection

- 2.4.1 The samples listed on the chain of custody (COC) are compared to the received samples to ensure all samples listed on the COC were received by the laboratory.
- 2.4.2 The samples and shipping coolers are inspected for leakage or breakage.
- 2.4.3 The temperature of the samples upon receipt is recorded.
- 2.4.4 The sample preservation is verified.
- 2.4.5 Any breach of the sample integrity will be noted and become a part of the project record.

2.5 Chain of Custody (COC) Verification

- 2.5.1 The COC establishes a traceable, legal record of the possession of the samples from sampling through analysis.
- 2.5.2 Laboratory personnel will compare the sample identification as listed on the COC to the identification on the samples.
- 2.5.3 Identify any sample requiring analyses that have short holding times and notify laboratory personnel of the sample arrival.
- 2.5.4 All discrepancies will be noted and become part of the project record.
- 2.5.5 Sample custody is maintained by storing the samples in a locked cooler.
- 2.5.6 Movement of the samples in and out of the cooler is tracked in a logbook.

2.6 ESAT LIMS Sample Log-In - After completing the sample receipt procedures, the samples are logged into the ESAT LIMS by utilizing the ESAT project definition and either an electronic XML file or by hand entering sample information from the COC and any noted discrepancies.

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3.0 ACRONYMS AND DEFINITIONS

°C	Degrees Celsius
COC	Chain of Custody
DM	Dissolved Metals
ESAT	Environmental Services Assistance Team
ID	Identification
LIMS	Laboratory information (data) management system
PO	Project Officer
PPE	Personal protective equipment
QA	Quality Assurance
QC	Quality Control
SOP	Standard operating procedure
TDF	Technical Directive Form
TR	Total Recoverable (Metals Fraction)
USEPA	United States Environmental Protection Agency

4.0 HEALTH AND SAFETY

4.1 The receiving personnel shall ensure the sample login area is clean and free of any potential contaminants prior to working in the area.

4.2 Proper personal protective equipment (PPE) is required for receipt of samples. This includes gloves, eye protection, and a lab coat at the minimum.

4.3 Leaking containers can pose a health risk due to the possible presence of acids, toxic components, etc., making inhalation of toxic vapors a possible hazard.

4.3.1 All coolers should be opened in a room with adequate ventilation.

4.3.2 If broken sample containers are present, additional PPE and engineering controls, e.g. fume hood, may be required. If the use of spill cleanup material is necessary, the proper method of cleanup and disposal must be followed.

4.3.3 Assistance from the ESAT or EPA Health & Safety Officer for proper handling and disposal procedures may be required.

4.4 Sample receipt personnel should be familiar with the location of additional safety equipment.

4.4.1 Spill and neutralizer equipment are available in the sample receipt area.

4.4.2 Eye wash and safety shower in the sample receiving area should be verified as unobstructed prior to unpacking the samples.

5.0 PERSONNEL QUALIFICATIONS

5.1 ESAT Team Members

5.1.1 The receiving and checking of incoming samples must be performed by an Environmental Services Assistance Team (ESAT) member trained in the proper performance of this SOP.

5.1.2 The sample receiver must be familiar with interpreting COC documentation, performing pH determinations, and maintaining custody of samples.

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5.1.3 Personnel receiving samples should have a baseline physical examination performed prior to receiving samples.
5.1.4 Some lifting of 30-50 pound coolers/containers may be required.

5.2 EPA Personnel
5.2.1 Periodically, EPA personnel will move coolers containing samples for ESAT into the ESAT sample storage cooler.
5.2.2 EPA personnel will notify ESAT team members of the arrival of the samples.

6.0 APPARATUS AND MATERIALS

6.1 Calibrated and Certified Thermometers
6.1.1 Thermometers used for measuring sample storage cooler temperatures are calibrated annually under the supervision of the EPA laboratory quality assurance officer using a certified thermometer.
6.1.2 An infrared temperature indicator is maintained and used by ESAT personnel for recording the temperature of the samples upon arrival.

6.2 Wide-range pH Paper.

6.3 Waste container, properly labeled.

6.4 Promium ELEMENT Laboratory Information Management System (LIMS) for sample tracking and reporting.

6.5 Laboratory fume hood for opening sample coolers.

6.6 Refrigerated and secured sample storage cooler.

7.0 COOLER RECEIPT AND ACCEPTANCE

7.1 Sample Integrity Inspection
7.1.1 Generally, samples are received through the main entrance of the facility via Fed Ex or delivery from the sampling contractor.
7.1.1.1 Save the copy of the air bill associated with each cooler and place it in the project folder.
7.1.1.2 The method of delivery is noted in the “Remarks” section of the COC and later is indicated in the project when the samples are logged into the LIMS.
7.1.2 Retrieve a sample cart from the metals instrument room (B-104) and move the coolers to the sample receipt area in the facility.
7.1.3 Examine the shipping coolers for any damage or leaks and note their presence for inclusion into the project folder.
7.1.4 Open the cooler(s) while the cooler is located under the exhaust hood in the sample receipt area.
7.1.5 Remove the COC from the cooler.
7.1.6 Sign the “Received” section of each page of the COC and indicate the data and time of the receipt.

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Note: *Ideally, the sampler or customer should be present during the transition of the samples into ESAT custody including opening of the coolers and cross-checking of information, whenever possible.*

7.1.7 Unpack the cooler using the COC to organize the samples on the work table in the sample receipt area.

7.1.7.1 If any issues with sample integrity are observed (e.g., damage to the sample container, contamination, etc.), the analyst should discuss this in the case narrative so that data users are aware that the sample may have been compromised.

7.1.7.2 Any correspondence with and direction received from the EPA PO regarding a compromised sample should be received in writing via email, and that email should be included in the data package.

7.1.8 Temporarily place the ice or baggies filled with ice in the deep sink under the exhaust hood.

7.1.9 Using the IR thermometer, measure the temperature of the first unpacked sample. This temperature will be recorded into LIMS and original COC.

7.1.10 Inspect each sample container for damage or leaking and note any circumstance for inclusion in the project folder.

7.1.11 Verify the preservation of any samples that are indicated on the COC as having been preserved to a specific pH.

7.1.12 Hold a piece of wide range pH paper over the waste container and pour 3-5 mL of each sample over the pH paper and into the waste container.

7.1.13 If the sample is properly preserved, no further action is required.

7.1.14 Recap the sample container and proceed with the log-in procedure.

Note: *The sample aliquots used to check the pH must be disposed of in the metals waste container in room B-104.*

7.1.15 Improperly preserved samples will be properly preserved before placing into the storage cooler. Carefully note on the COC and case narrative which samples were not properly preserved.

7.2 Chain of Custody (COC) Verification

7.2.1 The samples should be accompanied by a COC, sample tags, and custody seals.

7.2.1.1 All information required on the forms and tags must be properly completed and legible.

7.2.1.2 The sample tag information must be verified against the corresponding sample information noted on the COC.

7.2.2 In the case of COC discrepancies, the sample ID Tag will be assumed as the true information and the discrepancies must be clearly noted on the COC with the login personnel's initials and date.

7.2.2.1 All COC discrepancies should be discussed in the case narrative of the data package.

7.2.2.2 If a COC discrepancy requires contact with the EPA PO, this should also be discussed in the case narrative of the data package. If COC discrepancies are resolved verbally with the EPA PO, an email should be sent to confirm the reconciliation of discrepancies and a copy of the email should be included in the data package with the COC.

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7.2.3 If the documentation is incomplete, the ESAT Manager and EPA PO will be contacted regarding the discrepancy. The EPA PO will decide if the process will continue.
7.2.4 After each sample is unpacked from the shipping container and the sampling information is verified, it is segregated into various storage trays by analysis.
7.2.5 The trays are labeled with a tag in a plastic shield with the project name, LIMS number, TDF number, due date, and requested analysis.
7.2.6 The labeled trays are then placed in the walk-in cooler "A" and secured by locking the cooler with the provided padlock.
7.2.7 The trays are removed by the analyst prior to analysis.
7.2.8 The analysts record the removal of the samples from the cooler by using the logbook in the sample receipt area.
7.2.9 Empty the plastic bags filled with ice that were placed in the sink and put the empty bags into the provided waste container in the sample receipt area.

8.0 PROJECT CREATION AND SAMPLE ENTRY IN LIMS

8.1 ESAT Project Creation in LIMS
8.1.1 Open the LIMS software.
8.1.2 In the Project Management dropdown menu, select "Projects".
8.1.3 Highlight a similar project and be sure to check it has the required test codes.
8.1.4 Select the "Copy" option.
8.1.5 Double click the "Superfund" client option.
8.1.6 Rename the project in the dialog window.
8.1.7 From the new project screen select "Edit".
8.1.8 Put the TDF number in both the "Project Number" and "PO number" fields.
8.1.9 Select the "project Manager" from the drop down menu.
8.1.10 Check that the default EDD is "StdESATExel_rev1.exe".
8.1.11 Enter the appropriate project name in the comments field.
8.1.12 If the test codes for the new project need to be changed, double click on "Test Codes" and from the drop down menu select the correct test codes for this project.
8.1.13 Save the project.

8.2 Work Order Creation in LIMS
8.2.1 From the "Sample Control" menu select "Work order".
8.2.2 Select "Import" and select the file location of the XML/Scribe file from the drop down menu.
8.2.3 Click the "Import" button.
8.2.4 From the "Analysis" tab match the appropriate test codes.
8.2.5 From the "Matrices" tab match the sample preservatives.
8.2.6 From the "Container" tab select "Default".
8.2.7 Click "Done" and the new work order screen will appear.

8.3 Work Order Information Editing
8.3.1 Select the Work order from the dropdown menu and click "Edit".
8.3.2 Select the project from the drop down menu in the top right corner.
8.3.3 The Project number and the PO number should match the TDF for the project.
8.3.4 In the "Submitted By" window select the appropriate sampler from the drop down menu.

8.3.5 In the “SDG Identifier” window, type in the TDF number.
8.3.6 In the “Shipped By” window select either “Walk in” or” Fed Ex” from the drop down menu.
8.3.6.1 If shipping was by Fed Ex, enter the tracking number in that window.
8.3.7 Select the turn around time to calculate the appropriate due date for the project.
8.3.8 Check the appropriate “Condition” boxes for the samples received.
8.3.9 Ensure the Analysis Test Codes are accurate and add/delete as needed.
8.3.10 Save the Work order.

8.4 Editing Samples in the Work order
8.4.1 Click on the “Samples” tab and “Edit”.
8.4.2 Verify that the sample name, container, location and comment (EPA Tag #) are correct.
8.4.3 In the “Report Matrix” drop down window select the one listed on the COC.
8.4.4 In the “Sample Type” drop down window select “Field Sample”.
8.4.5 In the “Sampled By” drop down window select the one listed on the COC.
8.4.6 In the “Work Analysis” windows the test codes may or may not be applied. If more tests are needed, click the “Work Analysis” tab to see all of the available test codes.
8.4.7 Repeat sample entry/editing for all samples in the project.
8.4.8 Save the work order and click the printer icon to print the sample information.

8.5 Project Folder Creation
8.5.1 Master Project Folder
8.5.1.1 Label a new folder with the Work Order number, Project Name, TDF Number and Due Date.
8.5.1.2 Place the original COC, TDF and the shipping label in this folder.
8.5.1.3 Place the LIMS printout of the samples entered in this folder.
8.5.1.4 Place any E-mail or other documents pertaining to the project in this folder.
8.5.1.5 All analytical data will be placed in this folder until final report generation.

8.5.2 Analytical Folder
8.5.2.1 On the LIMS PC go to ”Explore”.
8.5.2.2 Go to the “X” drive and click on “Metals_Data_Files”.
8.5.2.3 Select the appropriate year.
8.5.2.4 Go to “File”, “New”, “Folder”.
8.5.2.5 Name the new folder using “Work Order_TDF Project Name” format. (Ex: (C606006_SC010 CalGulch June Monthly)
8.5.2.6 Repeat the file creation sequence in “WetChem_Data_Files” if the project requires this type of analysis.

8.5.3 Reporting Folder
8.5.3.1 On the ESAT PC, click and open “Explore”.
8.5.3.2 Go to the “G” drive and click on “ESAT”.
8.5.3.3 Click on “Analytical Reports” and then “Final Reports”.
8.5.3.4 Go to “File”, “New”, “Folder”.
8.5.3.5 Name the folder using the same convention as the Analytical Folder.

9.0 DATA AND RECORDS MANAGEMENT

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9.1 The sample checkout logbook is maintained by the EPA quality assurance/quality control QA /QC personnel.

9.2 Full logbooks will be archived, and new ones provided when necessary.

9.3 The EPA QA/QC personnel also verify thermometer calibration and log the Cooler temperatures daily.

9.4 COC records, LIMS reports, and all other correspondence become part of the ESAT retained records data file.

9.5 All entries to the sample checkout logbooks and custody records will be recorded in blue or black indelible ink.

9.6 When an entry error occurs, the author will draw a single line through the error, initial and date it, and complete the correct entry. If the space is too small for further legible entries, either the next line will be used, or the correction must be footnoted to ensure legibility of the correct entry.

9.7 Annual audits will be conducted by the ESAT QA/QC Coordinator to verify the procedures outlined in this SOP are being performed.

9.8 Refrigerated cooler temperatures are checked and recorded daily according to EPA Region 8 SOP Monitoring Refrigerator and Cooler Temperatures, current version.

10.0 WASTE MINIMIZATION

10.1 The analyzed samples are separated for consolidation and disposal. Consult the Aqueous Corrosive Waste Disposal SOP for further details (LAB-01.00_ESAT Waste Disposal_R2).

10.2 Plastic sample tag holders are reused, as are the washable trays, coolers, and carts. Sample containers are too easily contaminated and are not reused.

10.3 In order to minimize contamination of large volumes of liquids, compatible samples marked for disposal will be consolidated without further dilution.

10.4 Field coolers and some packing materials are cleaned, dried, and reused.

11.0 REFERENCES

11.1 USEPA Region 8 Laboratory SOP, Sample Receipt and Custody, current version.

11.2 ESAT Region 8 SOP, Disposal of Aqueous Corrosive Wastes.

11.3 ESAT Region 8 Health and Safety Plan, Current Version.

11.4 USEPA Region 8 Laboratory Chemical Hygiene Plan, Current Version.

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Technical Approval: ____Date:____________

QA Management Approval: ___________________________________Date:__________

SOP Description

This Standard Operating Procedure (SOP) describes the process to be followed by TechLaw staff when acquiring analytical support. All requests for analytical services are to be arranged through the Laboratory Assistance Team (LAT) Coordinator, or alternatively through a LAT member (see Attachment A for a list of approved LAT members). This SOP is to be followed by the TechLaw project manager or designee when completing and submitting the Analytical Support Request Form (ASRF) (see Attachment B), and after the sampling event is completed by submitting copies of the chain-of-custody forms to the LAT and forwarding invoices to the project files. A LAT member checklist is provided in Attachment D and a TechLaw Project Manager checklist is provided in Attachment E.

This SOP is also to be followed by the LAT Coordinator and assigned LAT members when processing the ASRF, selecting a laboratory and reviewing data packages and invoices.

General Procedures

Related SOPs

This SOP is to be used in conjunction with other applicable SOPs found in the following SOP categories:

Category No.	Category Title
01	General Procedures
02	Field Procedures
03	Field Documentation Procedures
04	Packaging and Shipping Procedures
05	Field Equipment Operation and Maintenance Procedures
06	Groundwater Sampling/Monitoring and Analysis Procedures

07	Soil/Sediment Sampling and Analysis Procedures
08	Surface Water Sampling and Analysis Procedures
09	Health and Safety Procedures
10	Regulatory Compliance Procedures
11	Quality Assurance Procedures
12	Incineration/BIF Sampling and Analysis Procedures
13	Waste Sampling and Analysis Procedures
14	Asbestos Handling
15	Region 5 ESAT-Specific SOPs
16	Region 8 ESAT-Specific SOPs

Procedures for Submitting and Processing the Form

The ASRF (see Attachment B) must be submitted to the assigned LAT member at least five business days prior to the sampling event to avoid additional charges for rush shipping of any necessary supplies. If supplies are not required from the laboratory, the ASRF may be submitted at least three business days prior to the sampling event. If shorter turnaround time is required, every effort will be made by the LAT to process the request. Faxed, hand-written requests or electronic requests are acceptable as long as they are legible and complete.

The assigned LAT member will ensure that all necessary information is included in the ASRF, and make necessary arrangements with the laboratory (e.g., request delivery of glassware or other sample collection media and equipment). The assigned LAT member will procure sample containers on an as-needed basis, as indicated on the ASRF.

Upon completion of the ASRF by the assigned LAT member, a copy is uploaded to the project folder under LAT on SharePoint. All samples must be sent to a TechLaw-approved laboratory. The laboratory is selected on the ability to perform the requested analysis, availability of laboratory space, and analysis cost. A Work Authorization (Attachment C) is submitted to the selected laboratory by the assigned LAT member prior to sampling activities. The Work Authorization is also e-mailed to TechLaw contracting staff for generation of a purchase order (PO). A copy of the finalized Work Authorization is placed in the project folder under LAT on SharePoint.

Procedures for Completing the Form

The ASRF (see Attachment B) must contain complete sampling and analytical information. This information shall include the following:

- Project number (billing code) and project manager name;
- Site name and location;
- Date(s) of sampling event;
- Required turnaround time;
- Type of data package (i.e., Level II, III or IV, requirements for summary forms);
- Special considerations, if any; and
- For the table: sample matrix; number of field samples; parameter (i.e., the appropriate analytical method numbers); required detection limits; and the numbers of quality control (QC) samples (i.e., field duplicates, trip blanks, field blanks, and matrix spike/matrix spike duplicates [MS/MSD]).

Procedures for Changes

If changes occur in the number of samples or type of sampling methods during the field activities, the project manager will notify the LAT member by email.

Procedures After Sampling Event Completion

Chain-of-Custody (COC) Forms

Within one week of shipment of samples (within 24 hours ideally), the project manager must ensure that legible copies of all COC forms have been sent to the assigned LAT member. Faxes or scanned e-mail copies are acceptable.

Data Package Review and Delivery

The laboratory sends all data packages directly to the assigned LAT member, who performs a preliminary review to ensure that the laboratory has submitted the requested information.

This review shall be performed within approximately 1 day of receipt. Each data package is evaluated by the assigned LAT member for the following criteria ONLY:

- Laboratory reports address methods specified on COCs.
- Requested results present for all samples.
- Appropriate level data package provided.
- Data received within requested turnaround time.

Upon completion of this review, the original data package is forwarded to the project manager, unless the project manager specifically requests otherwise.

Invoices

The laboratory sends all invoices to the assigned LAT member. Upon receipt of the invoice, it is reviewed for agreement with the Work Authorization and project sampling documentation. Upon confirmation (within approximately 24 hours of receipt), the LAT member will forward the invoice to TechLaw Accounts Payable and send a copy to the TechLaw Project Manager. A copy of the approved invoice is uploaded by the LAT member to the company intranet site, under LAT > Project Files.

Communication with the Laboratories

A member of the LAT, preferably the assigned LAT member, is to participate in all communications with laboratories. This is to ensure that all procedures required under the Laboratory Agreements and the TechLaw Quality Assurance Program Plan are met. The LAT member will notify the laboratory of any unusual situations, including the expected presence of high concentrations of contaminants at the site.

Obtaining New Laboratories

All samples must be sent to a TechLaw-approved laboratory. Names of approved laboratories may be obtained from the LAT Coordinator. Only senior members of the LAT, with the concurrence of the TechLaw Quality Assurance Director (QAD), may approve a new laboratory; this approval is conditional upon examination of laboratory-specific documentation. Necessary documentation consists of a laboratory quality assurance plan (must include a description and number of instruments, staff resumes and a summary of quality assurance/quality control procedures), SOPs, method detection limits (MDLs), quantitation limits, PE sample results and pricing.

A laboratory audit may also be performed by the TechLaw LAT. Audits will be performed for any laboratory that will be utilized on an ongoing basis. A laboratory audit may not be required if so directed by the client, or if only a specialty analysis is requested and the laboratory will not be used on a routine basis. The laboratory audit checklist is included as Attachment F to this SOP.

After a laboratory has been approved by the LAT, a Laboratory Agreement shall be arranged by the TechLaw Contracts Administrator, working in conjunction with the senior LAT member responsible for assessing the laboratory's qualifications. Only after these procedures have been completed may a new laboratory be used for analytical services.

The laboratory list is reviewed and updated annually, as necessary. LAT laboratories are contacted to obtain any updated laboratory documentation and pricing. Audits will be conducted at least every other year for laboratories performing routine analyses in the program.

Health and Safety

Not applicable

QA/QC

None at this time

Comments/Notes

Under no circumstances is it acceptable to provide the laboratory with the name, location or other identifying information for the site (this includes listing facility information on the chain-of-custody). Facility initials, TechLaw project number or other identifier should be used that will not reveal facility information to the laboratory, but will be evident to TechLaw employees involved with the project. If the laboratory becomes aware of the site name, the LAT member should inform the TechLaw COI Officer immediately. The COI Officer will ensure that the laboratory does not have a COI and will post documentation of this confirmation to the LAT project files on SharePoint.

The time required to arrange analytical services and process data packages and invoices will be charged to the appropriate project.

Attachments

Attachment A — Approved LAT Members

Attachment B — Analytical Support Request Form

Attachment C — Laboratory Work Authorization (generic)

Attachment D — LAT Member Checklist

Attachment E — TechLaw Project Manager Checklist

Attachment F — Laboratory Audit Checklist

References

TechLaw, Corporate Quality Management Plan, most current revision.

TechLaw, Health and Safety Program, most current version

TechLaw, Inc. Approved LAT Members

LAT Coordinator: Ms. Kim Whitlock
205 West Wacker Drive, Suite 1622
Chicago, IL 60606
(312) 345-8930
(217) 721-5483 (mobile)

LAT Members:

Ms. Rachel Ireland 7 Technology Drive, Unit 202 North Chelmsford, MA 01863 (978) 275-9749 (617) 283-1332 (mobile)	Ms. Zara Brown 14500 Avion Parkway, Suite 300 Chantilly, VA 20151 (703) 818-3213 (865) 898-3815 (mobile)
Mr. Gene Nance 5455 County Road 2 Chesapeake, OH 45619 (740) 867-0968 (304) 830-1442 (mobile)	Ms. Amy Dahl 1325 4th Ave, Suite 555 Seattle, WA 98101 (206) 826-5375 (206) 818-8891 (mobile)
Mr. Scott Walker 16194 W. 45th Dr. Golden, CO 80403 (303) 312-7726 (303) 453-9018 (mobile)	

If none of the above LAT Members are available, and an urgent laboratory need/data issue arises, please contact:

Ms. Jana Dawson
14500 Avion Parkway, Suite 300
Chantilly, VA 20151
(703) 818-3254
(703) 627-0821 (mobile)

Mr. Terry Zdon
1299 Washington Ave, Suite 270
Golden, CO 80401
(303) 552-5807
(773) 343-8974 (mobile)

TechLaw COI Officer: Ms. Judy Manley
14500 Avion Parkway, Suite 300
Chantilly, VA 20151
(703) 818-3233
(703) 209-5187 (mobile)

ANALYTICAL SUPPORT REQUEST FORM

Project Number (Bill Code): ___________________ TechLaw Project Manager: ________________

Site Name and Location: ____

Site Code (identifier)*: ____

***Site Code should not reveal the site name or location and will be the only identifier used in communication with the laboratory (including COCs) to prevent COI issues**

Example: Site Name and Location: Buckeye Products in Adrian, Michigan
Site Code: BAM (used the first letter of facility name, city, and state)

Date(s) of Sampling Event: ____

Glassware: Date Needed: ___________________ Location: ____________________________

Turnaround Time (circle one): Standard (21 days) / Rush-_____ days (extra charge)

Data Package: Level IV (full "CLP-like") / Other (Level II or III)___________________

Electronic Date Deliverable? Yes / No Format (i.e., Excel): ______________________

Special Considerations:

- **Are any special certifications required?**
- **Are there minimum volume or filter requirements?**
- **Is there a specific QAPP requirement?**
- **Are high concentrations expected?**
- **Are verbal or preliminary results required?**

PLEASE ATTACH A TABLE OF THE APPLICABLE SCREENING LEVELS FOR ALL COMPOUNDS TO BE ANALYZED. The LAT staff will use this information to verify that the laboratory reporting limits will meet the specified screening levels.

Matrix [1]	Number of field samples	Parameter (method #'s)[2]	Required Reporting Limits	Number of field duplicates	Number of trip blanks	Number of field blanks	Number MS/MSD

1 Be specific (i.e., surface H2O, liquid fuel, slag; not just solid/water).

2 Be specific; if split sampling, attach applicable MDLs, action levels, methodologies, etc., from facility work plan. For metals, please specify which compound list should be utilized: RCRA 8, priority pollutants, Target Analyte List

Note: Target Analyte List compounds will be utilized, unless otherwise specified by the Project Manager.

TechLaw Project Manager Signature: ___________________ Date: ________________

LAT Signature:_______________________ Date: ____________________

ATTACHMENT C [Revised 04/27/12]
SOP Number: 02-06-05

Date:

To: Contact
Laboratory
Address
Address

From: Kim Whitlock, LAT Coordinator
205 West Wacker Drive, Suite 1622
Chicago, IL 60606
(312) 345-8930
(312) 345-8979 (fax)

Re: EPA Prime Contract XXXXX
TechLaw Laboratory Agreement
Task Order Authorization Number: XXXX
Site Code (identifier): XXX
Project Code: [Insert billing code]

This document authorizes work on the subject Task Order as outlined in the attached Scope of Work and Pricing quotation (Attachment A). The expenditure limits on the Task Order are XXXXX. If it is anticipated that these funding limitations will be exceeded in performance of this work, you must notify us in a timely manner. Failure to notify and negotiate additional funding will result in forfeiture of costs incurred in excess of the funding limitations. Invoices should be sent to the Laboratory Assistance Team (LAT) representative noted above.

Please acknowledge your acceptance of work by signing in the space provided on the form, faxing a signed copy of this Task Order Authorization Form to Brenda Smith at (703) 818-8813, and returning the original within 5 days of your receipt to [LAT member name]. By acceptance of this Work Authorization, the Laboratory confirms that: no known personal or organizational conflict of interest exists; best efforts will be employed to conduct the work specified to the satisfaction of TechLaw, Inc. representatives; all terms and conditions of the Agreement identified and the Scope of Work and Pricing document will be met in performance of the work specified herein.

Authorized Signatures:

TechLaw, Inc.	**Laboratory Name**
Name:	Name:
Title:	Title:
Date:	Date:

ATTACHMENT D [Revised 04/27/121]
SOP Number: 02-06-05

LAT Member Checklist

1. If a TechLaw project manager contacts you regarding laboratory procurement, please ask them to fill out the Analytical Support Request Form (ASRF) in Attachment B of SOP 02-06-05, if they have not already done so. Upload completed form to the project file on SharePoint.
    a. Be sure to ask if there are any reporting limit requirements, and verify with the laboratory that they can achieve such requirements
2. Contact three TechLaw Approved Laboratories to obtain price quotes and to ensure they have capacity to analyze the samples within the requested turnaround time.
    a. If a specialized analysis is required and is not performed by a TechLaw Approved Laboratory, another laboratory may be used upon consultation with the LAT Coordinator.
3. Select the laboratory based on lowest pricing and ability to perform the requested analyses.
4. Fill out the Work Authorization Form in Appendix C of SOP 02-06-05, PDF file, and attach the analytical quote to end of the PDF. Submit the form to the selected laboratory via e-mail (copy Ms. Brenda Smith and Ms. Judy Manley). Ms. Smith will email the LAT member the PO associated with the analytical request.
5. Check the appropriate laboratory folder under LAT on SharePoint to ensure we have the SOP for the methods requested for the project. If these are not available already on SharePoint, request a copy of the SOP from the lab and upload to the laboratory folder on SharePoint.
6. Create a folder for your project under LAT > Project Files.
7. Update the Project Tracking spreadsheet under LAT > Project Tracking and ensure justification for laboratory selection is included in the Comments column.
8. Upon submission to the laboratory, upload a copy of the Work Authorization Form (unsigned by lab) to LAT > Project Files > Project Name.
9. Once a signed copy of the Work Authorization Form is received from laboratory, upload to LAT > Project Files > Project Name.
10. Confirm with laboratory when/where to send bottleware.
11. Inform the laboratory when to expect samples (it is also a good idea to remind them one day before they will receive samples).
12. Ensure the TechLaw field team submits a copy of the COC to you for verification of invoicing/sample data.
13. Upon receipt of the analytical data, review for the following:
    a. Laboratory reports address methods specified on COCs
    b. Requested results present for all samples submitted to the lab
    c. Appropriate level data package provided
    d. Data received within the requested turnaround time
    e. Scan through the laboratory case narrative for any major issues that would result in rejection of data.
14. If the items above are acceptable, approve invoice by signing, dating, and adding the proper PO number (including line item number) and forward to Patti Pinkard.
15. Upload a copy of the approved invoice to the project folder under LAT > Project Files.
16. Email a copy of the approved invoice to the TechLaw project manager.
17. Ask the TechLaw Project Manager where the data should be sent, and send out ASAP. If electronic data is available, upload a copy to the project folder under LAT > Project Files

ATTACHMENT E [Revised 04/27/12]
SOP Number: 02-06-05

TechLaw Project Manager Checklist for LAT

1. Fill out the Analytical Support Request Form in Attachment B of SOP 02-06-05.
2. Notify LAT member of any changes in schedule/requirements as soon as possible.
3. Ensure Field Team provides a copy of completed COCs to LAT member.
4. Inform LAT member where to send data upon receipt.

LAT members will:

- Procure laboratories
- Order bottleware, preservatives, and laboratory-grade water for blanks (if requested to do so)
- Handle all communication with the laboratory
- Add another laboratory to the TechLaw Approved Lab List only if required by the project (i.e., 24-hour turnaround for an emergency response) and current approved laboratories cannot fulfill the project requirements. Note: Additional time will be required to obtain the necessary information and add the laboratory to the approved list.

LAT members do NOT:

- Verify reporting limits if no requirements have been provided by the TechLaw Project Manager
- Perform data validation (unless qualified and asked to do so)
- Coordinate a data validator (unless asked to do so and authorized hours are provided)

LABORATORY AUDIT CHECKLIST

Laboratory On-Site Visits

There are several purposes for making on-site visits to analytical laboratories. The most common purposes are:

1. Prior to award of a contract or delivery of samples, a client visits the laboratory to verify that the laboratory has the capability to perform the needed work. The areas for which capability must be judged are:

    - Physical facility – adequate work space, adequate and appropriate air handling, adequate storage space.

    - Equipment – all equipment (instrumentation, reagents, glassware, etc.) needed to do the job at the needed frequency.

    - Personnel – trained, experienced personnel who meet the clients' requirements.

    - Standard Operating Procedures (SOPs) – written procedures must be in place (and updated when changes to "modus operandi" are made) for all operations of the laboratory so that consistency and continuity are maintained where appropriate.

    - Quality Assurance Program – includes all aspects of EPA's "Good Automated Laboratory Practices" (GALP) Guidance.

    - Appropriate evidentiary procedures, Chain-of-Custody documentation, and security systems must be in place.

2. Post contract award or after sample delivery by a client (at intervals determined appropriate by the client), the laboratory can be visited to verify that the capabilities evaluated in Number one (1) still exists, or improvements cited as needed or deficiencies cited as requiring correction in Number one (1) have occurred.

3. Problem resolution visits – When problems are noted by the client (e.g., performance evaluation samples not analyzed acceptably, lateness, non-compliance with contract requirements, etc.) laboratories can be visited to isolate problem areas and identify where corrective action must be taken by laboratory management.

4. Unannounced visits – to verify that the laboratory follows procedures and maintains systems per client's requirements, even when the client's visit is not expected.

5. Unannounced visits – when there is reason to believe a laboratory may be involved in improper practices (e.g., data falsification/alteration), a client may want a "surprise" visit. This visit should focus an audit on the area perceived to be vulnerable.

6. Routine – even when a laboratory is performing well, a client presence to show interest (and maybe a "pat on the back") is important every other year at a minimum.

As we look at the cited purposes, we should take the opportunity to identify what we really need to do during an on-site visit to meet our needs. We should minimize the universe of possible targets for evaluation and focus on what is important.

We should recognize that the capability to meet our needs (produce our required product) may come in many forms, and a stereotyped approach on our part is unreasonable and unnecessary. If we accept this premise, we go a long way towards minimizing our efforts in auditing the laboratory and opening the door to innovation and creativity on the part of our laboratories that may save time and money and may produce a better product.

Since the first two types (and sometimes Number 6) of on site visits are the most common and involve looking at the same things (which are primarily amenable to a checklist approach), the first effort at a new design of an on-site visit will consist of an appropriate checklist.

It is important to note that a checklist only meets part of our needs: one-on-one conversations with laboratory personnel who will perform our work should occur to make sure they understand our requirements, follow their SOPs, are properly trained, etc.

Proposed checklists for Technical and Evidentiary on site visits (audits) are attached. They are designed to be used together so one auditor can perform the full gamut of evaluation in one swing through a laboratory. The auditor should become familiar with the checklists prior to an audit so that all necessary checks can be made in one location in a laboratory at one time.

If items (e.g., SOPs) are addressed in the evidentiary section, then they are not addressed in the technical section. Since there is so much in common for organics and inorganics, the checklist is combined, with items unique to one or the other clearly identified.

These checklists are not contract specific. They can be used for non-CLP and CLP because they do not demand conformance, only observance of what is in place. The auditor is responsible for determining if the laboratory appears to meet the requirements of the client (which in the case of CLP labs, is the SOW).

The auditor should only evaluate the laboratory according to the items that the client considers relevant to meet needs (for CLP contracts, this is all items). Judgment should be used when determining what items are relevant to meet needs.

Laboratory On-site Visit Evaluation

Laboratory name:

Address:

Telephone number:

Date of in-site visit:

Type of evaluation 1) organics 2) inorganics 3) evidentiary

Evaluation Team Members

Name	Title	Evaluated 1, 2 or 3 above?

Personnel interviewed/contacted

Name	Title	Interviewed for 1, 2 or 3 above?

Evidentiary Procedures Evaluation Checklist

I. Sample Receipt

	Check one: Yes	No
1. Is there a designated sample custodian and alternate for each shift?	_______	_______

Names:

Sample custodians	Alternates
____________________	____________________
____________________	____________________

	Yes	No
2. Are standard operating procedures (SOPs) for sample receipt in place and readily available?	_______	_______
3. Are SOPs for sample receipt followed by laboratory personnel?	_______	_______
4. Is the sample receipt area secured against non-authorized personnel?	_______	_______
5. Does sample custodian verify the following:		
a. Condition of shipping cooler	_______	_______
b. Presence or absence of custody seals	_______	_______
c. Condition of custody seals, when present	_______	_______
d. Custody seal numbers, when present	_______	_______
e. Presence or absence of chain-of-custody record(s)	_______	_______
f. Presence or absence of airbill stickers	_______	_______
g. Airbill or airbill sticker number	_______	_______
h. Presence or absence of sample tags	_______	_______
i. Sample tag numbers (if applicable)	_______	_______
j. Condition of sample containers	_______	_______
k. Discrepancies in any information recorded on chain-of-custody records, client requests, airbills, sample containers, etc.	_______	_______

	Yes	No
l. Documentation of hand deliveries	_______	_______
m. Problems encountered	_______	_______
6. Obtain examples of all forms used during sample receipt	_______	_______

II. Sample Identification

	Yes	No
1. Does the laboratory have a unique sample identification system (i.e., vs. using client sample identification numbers)?	_______	_______
a. Is the number assigned upon receipt? If no when?_____________________________	_______	_______
b. If "yes", are numbers cross referenced to client numbers in a log?	_______	_______
2. Does the system clearly apply to samples, extracts, digestates, etc.?	_______	_______
3. Are SOPs readily available for sample identification?	_______	_______
4. Are SOPs for sample identification followed by laboratory personnel?	_______	_______
5. Obtain example of laboratory's sample identification system (e.g., example sample number with cross reference)	_______	_______

III. Sample Storage and Tracking

	Yes	No
1. Are sample (and extracts, etc.) storage areas secured and access to samples (extracts, etc.) available only to authorized personnel?	_______	_______
2. Are samples (extracts, etc.) logged in/out of storage area(s) when accessed?	_______	_______
3. Are samples (extracts, etc.) tracked throughout analytical process (e.g., a traveler sheet)?	_______	_______
4. Are SOPs for storage and tracking of samples (extracts, etc.) readily available?	_______	_______

	Yes	No
5. Are SOPs for storage and tracking followed by laboratory personnel?	_______	_______
6. Obtain examples of all forms/documents used for storage and tracking records	_______	_______

IV. Document Review

	Yes	No
1. Evaluate documents with the following questions in mind.	_______	_______
a. Are activities (e.g., GC/MS-VOA, ICP-metals) identified on all analysis documents?	_______	_______
b. Are titles on all documents?	_______	_______
c. Are columns labeled with headers?	_______	_______
d. Are reviewers' signatures identified when applicable?	_______	_______
e. Is the laboratory's name on all documents?	_______	_______
f. Are all entries fully dated (day, month, year)?	_______	_______
g. Are entries signed by the responsible person for performing and recording activities?	_______	_______
h. Are all logbook and other document entries in ink?	_______	_______
i. Is error correction protocol followed? (single line through area to be corrected and corrector's initials – no "white out")	_______	_______
j. Are pages in bound and unbound logbooks sequentially numbered?	_______	_______
k. Are log-book entries in chronological order?	_______	_______
l. Is inserted information taped into logbooks signed and dated when activity is performed?	_______	_______
m. Are unused portions of documents lined out?	_______	_______

		Yes	No
2.	Do documents provide a complete record of activities observed by the evaluator?	______	______
3.	Are instrument run logs maintained to enable a reconstruction of the run sequence on an instrument?	______	______
4.	Are records of failed runs maintained?	______	______
5.	Is disposal/depletion of samples (extracts, etc.) documented?	______	______
6.	Is data transferred electronically within the laboratory? If "yes", is a hard copy printed and retained in a client/case file?	______	______
a.	If data is transferred electronically, is the following information recorded?	______	______
1)	Person responsible for electronic data transfer?	______	______
2)	Date of electronic transfer?	______	______
3)	Person to whom data was electronically transferred?	______	______
4)	Status of electronically transferred data (e.g., draft final, etc.)?	______	______
5)	Numerical identifier assigned to electronic data transfer?	______	______
b.	Are SOPs readily available for electronic data transfer?	______	______
c.	Are SOPs followed by laboratory personnel for electronic data transfer?	______	______

V. Confidential Information

1.	If laboratory receives confidential information/documents, is a system set up to maintain that confidentiality, including for data generated on associated samples?	______	______

VI. Case (Client's Designated Group of Samples) File Organization and Assembly

Name:_______________ _____________________
Document Control Officer — Alternate

	Yes	No
1. Are case documents maintained in a secure area?	______	______
2. Is shipment of deliverables to clients documented?	______	______
a. Is recipient identified?	______	______
3. Are deliverables sealed with custody seals?	______	______
a. Are custody seals signed?	______	______
b. Are custody seals dated?	______	______
4. Does document control officer assemble and cross check information to assure that data on each case file is consistent and complete?	______	______

VII. Security of the Facility

1. Are visitors required to sign in?	______	______
2. Are visitors required to display distinct badges/ID?	______	______
3. Are all doors to outside locked except to reception area?	______	______
4. Is access to laboratory and data reduction/report preparation areas limited to authorized personnel?	______	______

Observations/Comments by Evaluators

I Sample receipt –

II Sample identification –

III Sample storage/tracking –

IV Document review –

V Confidential information –

VI Case file organization and assembly –

VII Security of facility –

Technical Procedures Evaluation Checklist
(Organics and Inorganics)

		Yes	No
I.	Sample Receipt and Storage Area (Evaluate with I-III of Evidentiary Audit)		
	1. Are sample shipping coolers opened in a contamination-free area (e.g., fume hood or vented area)?	______	______
	2. Are adequate facilities provided for the cold storage of samples and unused samples for 60 days after data submission?	______	______
	a. Is the temperature of the cold storage recorded daily in a logbook?	______	______
	b. Are temperature excursions noted and appropriate actions taken when required? (Check SOP)	______	______
	3. For inorganic only: Is the pH of the samples recorded and available for the data review?	______	______
	4. For organic only: Are volatile samples stored separately from semi-volatile samples and extracts?	______	______
	5. For organic only: Are VOA holding blanks present in the volatile sample storage facility? (One per case)	______	______
	6. For organic only: Are sample extracts properly stored (2-6oC, separate) and easy to locate by reference to a logbook?	______	______
II.	Sample Preparation Area		
	1. Is the laboratory maintained in a clean and organized manner appropriate for trace level analyses (contamination free)?	______	______
	2. Does the laboratory appear to have adequate work space (6 linear feet or unencumbered bench top/analyst)?	______	______

		Yes	No
3.	Are laboratory benches made of suitable chemically resistant materials?	_______	_______
4.	Are sufficient functional hoods available?	_______	_______
5.	Is documented organic free water (for organics standards, blanks, dilutions) or distilled/ demineralized water (for inorganics) available?	_______	_______
6.	Are analytical balances located away from drafts and areas subject to rapid temperature changes?	_______	_______
	a. Are the balances checked routinely (e.g., before each weighing session) with the appropriate range of weights and results recorded in a permanent notebook?	_______	_______
	b. Are routine weights checked against class S weights at least once a month and Results recorded in a permanent notebook?	_______	_______
	c. Have the balances been calibrated within one year by a certified technician?	_______	_______
	d. Are the data generated from balances electronically transferred or manually entered into LIMS?	_______	_______
7.	Are sample preparation SOPs readily available?	_______	_______
	- Are sample preparation SOPs followed by laboratory personnel?	_______	_______
8.	Are glassware preparation/cleaning SOPs readily available?	_______	_______
	- Are they followed by laboratory personnel?	_______	_______
9.	Is all required sample preparation equipment available?	_______	_______
	For organics only:		
	a. Sonicator	_______	_______

Make______Model________Backup_______(Y/N)

	Yes	No
b. GPC	_______	_______
Make______Model________Backup_______ (Y/N)		
c. GPC UV Detector	_______	_______
Make______Model________		
d. Do GPC logs indicate corrective actions are taken when there is a problem with calibration?	_______	_______
e. Continuous liquid/liquid extractors? Number_______	_______	_______
For inorganics only:		
f. If microwave digestion is to be used, is an adequate microwave ovens (programmable power setting up to 600 watts) available?	_______	_______
10. Do analysts record bench data in a neat and accurate manner?	_______	_______
11. Do analysts record lot number of solvents, spiking solutions, etc., on bench sheets?	_______	_______
12. Is there evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents?	_______	_______
13. Review the following procedures for oven drying:		
a. Are the temperatures in the drying ovens verified against NIST traceable thermometers?	_______	_______
b. Do ovens have temperature logbooks?	_______	_______
c. Are “in” & “out” drying times recorded?	_______	_______

III. Standards Preparation and Storage

1. Are SOPs for standards preparation readily available?	_______	_______

		Yes	No
2.	Are SOPs followed by laboratory personnel?	______	______
3.	Are reagent grade or higher purity chemicals used to prepare standards?	______	______
4.	Are standards properly labeled with concentrations, date of preparation, expiration date, and/or a traceable reference code number?	______	______
5.	Are spiking/calibration standards preparation and tracking logbooks maintained for:	______	______
	Inorganics?	______	______
	Semi-volatiles?	______	______
	Pesticides?	______	______
	Volatiles?	______	______
	- Are logbook numbers and series of stock Solutions and reagents recorded?	______	______
6.	If the laboratory purchases commercially prepared standard mixes, is appropriate documentation available? (Manufacturer's Certificate of Analysis)	______	______
7.	Inorganic only: If the laboratory uses automatic pipets for preparing standards, are they routinely calibrated?	______	______

IV. Analytical Instrumentation and Analyses-Specific Items

ORGANICS

		Yes	No
A.	GC/M's (for up 200 samples/month)	______	______
1.	Instrument needs	______	______
	a. 1 VOA GC/MS/DS with purge and trap device	______	______
	b. 2 semi-VOA GC/MS	______	______
	c. 1 Backup GC/MS/DS and purge and trap device	______	______

GC/MS/DS Instr ID	Manuf./model	Software rev.	Purge and Trap InstrID	Manuf/model

		Yes	No
2.	Are manufacturers' operating manuals readily available?	_______	_______
3.	Does laboratory have service contracts?	_______	_______
	a. Does lab have extensive replacement parts available?	_______	_______
4.	Is a permanent service record maintained for each instrument?	_______	_______
5.	Does the laboratory use a recent mass spectral library?	_______	_______
6.	Magnetic tape storage of GC/MS electronic data:	_______	_______
	a. Are raw data, including quantitative output files and libraries, archived on magnetic tape?	_______	_______
	b. Is a log of raw data contents of tapes maintained?	_______	_______
7.	VOA analyses:	_______	_______
	a. Is equipment available for heated purge and trap for low level soil analysis?	_______	_______
	b. Are VOA holding blanks results available?	_______	_______
8.	Can instrument operator show from the run log that corrective actions have been taken for:	_______	_______

		Yes	No
	a. Re-analyses when internal standard areas are out of control?	_______	_______
	b. Dilutions when calibration range is exceeded?	_______	_______
	c. Blanks when previous sample showed saturation?	_______	_______
9.	Are SOPs readily available for GC/MS analyses and logbook completion?	_______	_______
	Are they followed by the laboratory?	_______	_______
10.	Is there evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents?	_______	_______
B.	GC/EC's (for up to 200 samples/month)	_______	_______
1.	Instrument needs	_______	_______
	a. 2 GC/EC/DS with dual columns	_______	_______
	b. 1 backup GC/EC	_______	_______

GC/EC Instr ID	Manuf./Model	Detector	Data System Manuf./Model	Revision #

		Yes	No
2.	Are manufacturers' operating manuals readily available?	_______	_______
3.	Does the laboratory have service contracts?	_______	_______
	a. Does the lab have extensive replacement parts available?	_______	_______
4.	Is a permanent service record maintained for each instrument?	_______	_______

	Yes	No
5. Are SOPs readily available for GC/EC analyses and logbook completion?	_______	_______
a. Are they followed by laboratory personnel?	_______	_______
6. Is there any evidence of a secondary review of all documents by someone other than the person generating the document?	_______	_______

INORGANICS

A. ICPs (for up to 300 samples/month)

1. Instrument needs	_______	_______
a. 1 ICP	_______	_______

Instrument ID	Manuf./Model	Seq./Sim

	Yes	No
2. Are manufacturers' operating manuals readily available?	_______	_______
3. Does laboratory have service contracts?	_______	_______
4. Are SOPs readily available for ICP analyses and logbook completion?	_______	_______
Are they followed?	_______	_______
5. Are stock standards current?	_______	_______
6. Are calibration standards made from a ready made stock standard? Manufacturer?__________	_______	_______
Are they prepared at least monthly?	_______	_______

		Yes	No
7.	Have any of the instruments been modified?	_______	_______
	If "yes", which one and how?____________________		
8.	How will calibration intensity and gains be kept?____________________		
9.	Is a mass flow controller used?	_______	_______
10.	Is interference correction done automatically and are interelement correction factors determined on at least an annual basis?	_______	_______
11.	Is a permanent service record maintained for each instrument?	_______	_______

B. ICP-MS (for up to 300 samples/month)

		Yes	No
1.	Instrument needs	_______	_______
	a. 1 ICP-MS	_______	_______

Instrument ID	Manuf./Model	Seq./Sim

		Yes	No
2.	Are manufacturers' operating manuals readily available?	_______	_______
3.	Does laboratory have service contracts?	_______	_______
4.	Are SOPs readily available for ICP-MS analyses and logbook completion?	_______	_______
	Are they followed?	_______	_______
5.	Are stock standards current?	_______	_______
6.	Are calibration standards made from a ready made stock standard? Manufacturer?_________	_______	_______
	Are they prepared at least monthly?	_______	_______

		Yes	No
7.	Have any of the instruments been modified?	______	______

If "yes", which one and how?__________________
____
____

8. How will calibration intensity and gains be kept?____________________________________
____

		Yes	No
9.	Are reanalysis performed when internal standards are out of control?	______	______
10.	Is a mass flow controller used?	______	______
11.	Is interference correction done automatically and are interelement correction factors determined on at least an annual basis?	______	______
12.	Is a permanent service record maintained for each instrument?	______	______

C. Atomic Absorption (AA) Spectrometer

		Yes	No
1.	Instrument needs		
	a. 2 GFAA for up to 200 samples/month	______	______
	b. 4 GFAA for up to 400 samples/month	______	______

GFAAs

Instrument ID	Manuf./Model

		Yes	No
2.	Are element specific SOPs which list instrument conditions, background corrections, and required instrument sensitivity readily available?	______	______
	Are they followed?	______	______
3.	Are calibration results (i.e., sensitivity) kept in a permanent record to track instrument performance?	______	______

		Yes	No
4.	Are calibration standards prepared daily?	______	______
5.	Have any of the instruments been modified?	______	______
	If "yes", which ones and how?________________ ____		
6.	Is an autosampler used?	______	______
7.	Are EPA or instrument manufacturers matrix modifiers used?	______	______
	As: ____________________ Pb: ____________________ Se: ____________________ Tl: ____________________		
8.	Is a permanent service record maintained for each instrument?	______	______

D. Mercury analyzer – Cold Vapor AAs

		Yes	No
1.	Instrument needs.	______	______
	a. 2 mercury cold vapor AAs for up 300 samples/month.	______	______

Cold Vapor AA

Instrument ID's	Manuf./Model

		Yes	No
2.	Are SOPs readily available for analyses and logbook completion?	______	______
	Are they followed?	______	______
3.	Are calibration standards prepared daily?	______	______
	With the samples?	______	______
4.	Is an absorbance record kept to monitor sensitivity?	______	______
5.	Is a permanent service record maintained for each instrument?	______	______

E. Cyanide Distillation Apparatus ______ ______

1. Needs – 12 distillation apparatuses plus one photometer for up to 300 samples/month ______ ______

Photometer

Instrument ID's Manuf./Model

____
____
____

		Yes	No
2.	Are SOPs readily available for cyanide analysis?	______	______
	Are they followed?	______	______
3.	Is there a stock cyanide standard from a commercial source?	______	______
	If "no", made from KCN salt?	______	______
	Is standard titrated?	______	______
4.	Is the titrimetric manual or semi-automated colorimetric method used? Method:______________	______	______
5.	Is the pH of the samples recorded and available for review?	______	______
6.	Are samples checked for the presence of sulfide and chlorine?	______	______
7.	Is a service log maintained for the photometer?	______	______
V.	Data Handling and Review (GALP)	______	______
1.	Are data calculations spot checked by a second person?	______	______
2.	Do records indicate appropriate corrective action when QC criteria are not met?	______	______
3.	Do supervisory personnel review the data and QC results prior to submission?	______	______
4.	Are SOPs for data handling/review readily available?	______	______
	Are they followed?	______	______

		Yes	No
5.	Are data and file access user ID of file password protected?	_______	_______
6.	Are deliverables checked for completeness and accuracy? (Hard copy and electronic)	_______	_______
Resubmittals?		_______	_______
7.	Is the monthly data entry error rate determined and recorded?	_______	_______
8.	When changes to deliverables are required, are the changes properly documented? (Rationale, review, initials.)	_______	_______
9.	Are user manuals and operations/systems manuals available?	_______	_______
10.	Is a written software test and acceptance plan available for installation of system changes?	_______	_______

VI. Quality Assurance Internal Inspections

		Yes	No
1.	Is there an internal QA inspection procedure?	_______	_______
2.	Does the QA officer report to senior management?	_______	_______
3.	Are corrective actions documented?	_______	_______
4.	What kinds of internal audits are performed?	_______	_______
	a. Blind PE sample?	_______	_______
	b. Other:____________________ ____________________	_______	_______
5.	What kinds of QA records are kept?		
	a. PE sample results?	_______	_______
	b. Records of recoveries (extractions, etc.)	_______	_______
	c. Training/experience records of personnel?	_______	_______
	d. Method sensitivities?	_______	_______

		Yes	No
e.	Control charts for QC purposes?	______	______
f.	Other ____________________		

VII. Quality Assurance Plan (QAP)

		Yes	No
1.	Is a QAP available?	______	______
2.	Does it address the following?	______	______
a.	Organization and philosophy	______	______
b.	Facilities and equipment	______	______
c.	Document control	______	______
d.	Analytical methodology	______	______
e.	Data generation	______	______
f.	QA	______	______
g.	QC	______	______
h.	Corporate ethics policy	______	______

VIII. Standard Operating Procedures

		Yes	No
1.	Are SOPs available for the following (many already addressed earlier during the audit)?	______	______
a.	Evidentiary	______	______
b.	Sample Receipt and storage	______	______
c.	Sample preparation	______	______
d.	Glassware cleaning	______	______
e.	Calibration (balance)	______	______
f.	Calibration (instruments)	______	______
g.	Analytical procedures (for each system)	______	______
h.	Maintenance activities (for each system)	______	______
i.	Analytical standards	______	______
j.	Data reduction procedures	______	______
k.	Documentation policy/procedures	______	______
l.	Data validation/self inspection procedures	______	______
m.	Data management and handling	______	______

		Yes	No
IX.	Organization and Personnel Summary (See "Key Personnel" list – attached)	_______	_______
1.	Do personnel assigned to this project have the appropriate educational background to accomplish the objectives of the program?	_______	_______
2.	Is the organization adequately staffed to meet project commitments in a timely manner?	_______	_______
3.	Were all key personnel available? List those not present.	_______	_______

Name	Position

		Yes	No
X.	Laboratory Capacity		
1.	Does the laboratory have sufficient analytical instrumentation to analyze the needed number of samples?	_______	_______
2.	Does the laboratory have sufficient technical administrative personnel to deliver the number of needed analyses?	_______	_______
3.	Does the laboratory have an adequate sample and data tracking system to handle the needed number of analyses?	_______	_______

XI. Summary

Provide an overall evaluation of the laboratory's apparent technical capability to perform the needed work.

KEY PERSONNEL LIST

List the personnel assigned to the following functional positions, and put a check next to any unqualified personnel.

Laboratory Operations Personnel

Project Manager

Name:______________________________________
Responsible for overall aspects of contract/project: primary contract

Quality Assurance Officer

Name:______________________________________
Generally requires a Bachelor's degree in chemistry/science/engineering + 3 years' laboratory experience, including 1 year applied experience with QA principles and practices in an analytical laboratory.

Laboratory Manager

Name:______________________________________
Responsible for overall management of the laboratory.

Sample Custodian

Name:______________________________________

Laboratory Personnel – Organics

Organics Supervisor

Name:______________________________________
Generally requires Bachelor's degree in chemistry/science/engineering + 3 years' organics experience, including 1 year supervisory experience.

Sample Preparation Laboratory Supervisor

Name:______________________________________
Generally requires Bachelor's degree in chemistry/science/engineering + 3 years' laboratory experience including 1 year supervisory experience. Three additional years experience may substitute for education requirement.

GC/MS Operator
Name:________________________________
Name:________________________________
Name:________________________________
Generally requires Bachelor's degree in chemistry/science/engineering + 1 year GC/MS/DS experience or 3 years GC/MS/DS experience and GC/MS interpretation. Three additional years experience may substitute for education requirement.

GC/EC Operator

Name:________________________________
Name:________________________________
Name:________________________________
Generally requires Bachelor's degree in chemistry/science/engineering + 1 year GC/EC experience or 3 years GC/EC experience and GC/EC interpretation. Three additional years experience may substitute for education requirement.

Extraction Concentration Expert

Name:________________________________
Name:________________________________
Generally requires High School diploma and college level course in general chemistry + 1 year experience in extraction/concentration.

Backup Chemists (Technical Staff Redundancy)

Name:________________________________
Generally requires Bachelor's degree in chemistry/science/engineering + 1 year lab experience in GC/MS operation, MS interpretation, extraction, and pesticide analysis.

Comments:

<u>Laboratory Personnel – Inorganics</u>

Inorganic Laboratory Supervisor

Name:________________________________
Generally requires a BS or BA in science. 1 year related experience, including 1 year as a supervisor.

ICP/ICP-MS Operator

Name:________________________________
Name:________________________________
Generally requires a BS or BA in science, 1 year ICP experience, 3 years additional experience in lieu of the education requirement.

Lachat Operator

Name:____________________________________
Name:____________________________________
Generally requires a BS or BA in science, 1 year Lachat experience, 3 years additional experience in lieu of the education requirement.

AA/Mercury Operator

Name:____________________________________
Name:____________________________________
Generally requires a BS or BA in science, 1 year experience for each of the following AA techniques: flame, graphite furnace, and cold vapor.
3 years additional experience in lieu of the education requirement.

Inorganic Sample Preparation Specialist

Name:____________________________________
Name:____________________________________
High school diploma and college level course in chemistry and 1 year related experience: 6 months experience plus 6 months experience with microwave digestion, if microwave technique is used.

Wet Chemistry Analyst

Name:____________________________________
Name:____________________________________
Generally requires a BS or BA in science and 1 year experience; 3 additional years experience may substitute for education requirement.

Attachment A
Black Chain of Custody Form

TechLaw
ESAT Region 8 Laboratory
16194 W 45th Drive
Golden, CO 80403
303.312.7047

US EPA CLP
Chain-of-Custody

EVENT: eCOC Template

Sample #	Tag	Location	Sub Location	Sample Type	Collection	Matrix	Analyses	Preservation	Sample Date	Sample Time	Remarks	Sampler

Relinquished By (DATE/TIME):

Received By (DATE/TIME):

Sampling/Analysis Notes:

Cooler Temp:__________
ICE: Y N
pH: Y N
Cust. Seals: Y N
COC/Labels Agree: Y N
Containers Intact: Y N

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16194 W 45th Drive
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US EPA CLP
Chain-of-Custody

EVENT: eCOC Template

Sample #	Tag	Location	Sub Location	Sample Type	Collection	Matrix	Analyses	Preservation	Sample Date	Sample Time	Remarks	Sampler

Sampling/Analysis Notes:

Relinquished By (DATE/TIME):

Received By (DATE/TIME):

Cooler Temp:__________
ICE: Y N
pH: Y N
Cust. Seals: Y N
COC/Labels Agree: Y N
Containers Intact: Y N

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Chain-of-Custody

EVENT: eCOC Template

Sample #	Tag	Location	Sub Location	Sample Type	Collection	Matrix	Analyses	Preservation	Sample Date	Sample Time	Remarks	Sampler

Sampling/Analysis Notes:

Relinquished By (DATE/TIME):

Received By (DATE/TIME):

Cooler Temp:__________
ICE: Y N
pH: Y N
Cust. Seals: Y N
COC/Labels Agree: Y N
Containers Intact: Y N